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AUTHOR(S) S. C. Stotlar, R. W. Springer, B. Sherwood, R. Cordi

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Los Alamos National Laboratory
Los Alamos, New Mexico 87545

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S. C. Stotlar, R. W. Springer, B. Sherwood, R. Cordi

Los Alamos National Laboratory
Materials Science and Technology Division
P.O. Box 1663
Los Alamos, New Mexico 87545

ABSTRACT

The efforts of the FEL source have been concentrated on cesiated GaAs(100) wafers. These crystals have shown photoyield of <.1 - 9 percent quantum efficiency with the cesium and oxygen treatment. The work function and coverage curves exhibit the same properties as measured in the literature. The use of Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy have been instrumental in determining the surface cleanliness and the surface oxidation states. The sputtered surfaces have been investigated as a function of rare gas mass and sputter ion voltage, giving similar results to earlier literature values. Temperature annealing appears to be critical after sputter cleaning in achieving any significant photoyield. Contacts of Ag-Mn and Ni-Si have been deposited, heated, and analyzed using Auger Depth Profiling techniques.

Introduction

The Free Electron Laser works on principles that can be explained solely using classical physics. A magnetic field formed by a series of alternating pairs of magnets creates a vertical magnetic field whose sign changes periodically along the array. The period is determined by the pair separation. In a typical wiggler, the field changes sign a few hundred times. The transverse oscillations of a beam of "free"* electrons moving through the wiggler will yield electromagnetic radiation which appears at the wiggle frequency to an observer moving with the electrons. In the laboratory frame of reference, this radiation is strongly forward directed and doppler shifted to a shorter wavelength

$$\lambda_L \sim \lambda_W / \gamma^2 \quad (1)$$

where λ_W is the period of the wiggler and γ is the electron energy in units of rest mass (.511 MeV). For example, a period of 2 cm. and 100 MeV electrons would produce light of wavelength 522 nm. Radiation of this type is known as magnetic Bremsstrahlung, synchrotron or undulator radiation. If an optical beam is superimposed on the electron beam and traveling in the same direction, the electrons will oscillate in phase with the incident beam when the incident beam is resonant with the electron oscillations. Resonance occurs when the wavelength of the incident beam equals λ_L . When the electrons oscillate in phase with the incident beam, their emission becomes coherent with respect to the incident beam and amplifies it as in a conventional laser. The energy added to the optical beam is extracted from the electron beam. Therefore, electrons emerge from the wiggler with less energy than when they entered. The Free Electron Laser offers the advantage of accessible wavelength, power scalability, and optical beam quality.

Many experiments of interest using the FEL require short laser pulses and focusing to small spots. Semiconductor negative electron affinity (NEA) photoemitters permit short wavelength excitation (<1.3 eV) and do not require bunching. NEA cathodes offer low emittance (low thermal spread in emitted electron energies) and high quantum efficiencies as well as potentially superior beam quality when compared to thermionic emitters. Conversely, they require ultra high vacuum pressures, they may be subject to dislocation damage from positive ions, and high quality materials may be difficult to obtain in the required sizes. Also, the NEA effect is caused by one to two layers of cesium with some oxygen present on the surface. Any contamination such as additional oxygen or additional cesium reduces the peak quantum efficiency.

* Not bound in atoms or molecules.

Background

Although electron emission into vacuum was observed nearly a century ago,¹ negative electron affinity devices were first reported in 1963.² One reason for the long time period between these two events is probably due to the lack of very good vacuum environments, 10×10^{-10} Torr (1.33×10^{-8} Pa). Another reason is that quality p-type semiconductors were not available until this time. The operation of NEA devices has been described in a variety of ways.³⁻⁵ Simply put, electrons can be excited from the valence band to the conduction band in emitters by light or high energy primary electrons. Only those electrons which reach the surface with energy X (the electron affinity*) can escape. However, if the electron affinity is made negative, transport to the surface is via minority carrier diffusion and the escape depth is the minority carrier diffusion length or the penetration depth of the exciting source, whichever is less. Higher emission efficiencies and lower emission velocities than can be obtained from conventional thermionic (hot electron) sources result. The energy distribution of the emitted electrons is narrower because most of them come from the bottom of the conduction band. If a semiconductor material is doped p-type, the valence band will coincide with the Fermi level and the conduction band will be above the valence band by a distance equal to the band gap. If the band gap E_g is greater than the work function ϕ , there is a truly negative electron affinity. In general, the bands will bend down near the surface. The position of the Fermi level at the surface is controlled by the surface states and in the bulk by the doping. The degree of band bending is determined by the magnitude of the doping. In order for electrons which have diffused to the bent-band region to cross, the region must not be significantly wider than the mean free path for hot electron energy loss. Therefore, the doping must exceed about 10^{17} acceptors/cm³. As the doping level increases, the bent-band region becomes narrower, increasing the probability of escape. However, the probability of an electron recombining with one of the acceptors increases and must be considered for each material. The critical part of the surface preparation is to lower the work function, i.e. surface barrier to below the bottom of the conduction band. Thus electrons with any kinetic energy that reach the surface may leave unimpeded. The depth of the band bending depends on the amount of p-type doping used. In general, the bands should be bent over 2 absorption depths so that 97% of the light entering the crystal promotes electrons that see the bent bands and are swept to the surface. The mathematical description of the quantum efficiency as a function of the diffusion length (semiconductor properties) and of the cesium overlayer (surface escape probability) is shown in equation 2:

$$\frac{(1 - R_f)}{QE} = \frac{1}{P} + \frac{1}{PL_{ac}} \quad (2)$$

where QE is the quantum efficiency, R_f the reflected fraction, P the escape probability, L the diffusion length, and α the absorption coefficient. Values of QE given in this paper are per incident photon and not per absorbed photon.

Better emission efficiencies and lower emission velocities can be obtained from the photocathode than achieved from a thermionic source. The energy distribution of the emitted electrons is narrower because most of them come from the bottom of the conduction band.

Lowering the work function can be accomplished by depositing a layer of any of the alkali metals on the atomically clean surface. Cesium appears to be the best candidate and is widely used to lower the work function of semiconductor surfaces. For gallium arsenide, the work function decrease is about 3.5 volts. Some small oxygen chemisorption can further lower the work function by another .6 eV. Turnbull and Evans⁶ originally suggested the use of the cesium-oxygen treatment for the III-V materials. The use of other gases such as fluorine has been used by other workers with success.

Two models are used to describe the surface escape probability. In the dipole model⁷, the cesium is ionized on the surface as the work function of the gallium arsenide is greater than the first ionization potential of the metal. The cesium deposited on the surface becomes ionized and forms dipoles on the surface. The resultant electric field lowers the work function in a linear fashion until the dipoles can no longer be screened from each other by the surface electrons. The addition of more cesium no longer lowers the work function and permits the dipoles to become depolarized and no longer perpendicular to the

*The work function ϕ of a crystal surface is the amount of energy required to move an electron from the crystal to infinity. The energy required to allow an electron to escape from the bottom of the conduction band is the electron affinity X . For a metal, $\phi = X$. Both are measured from the Fermi level. For a semiconductor, the electron affinity is measured from the bottom of the conduction band to the vacuum level and is a constant for a given semiconductor. The work function is measured from the Fermi level to the vacuum level and depends on the doping and on the kind of crystal.

surface. The addition of some oxygen to the surface causes surface reconstruction by moving between the cesium and the surface. This layering is thought to increase the polarization and further lower the work function. Any additional oxygen will reside on the surface, thus placing the electro-negative element on the surface and causing an increase in the work function. A schematic of this model is shown in Fig. 1a.

Another theory for the escape probability that has been suggested by Spicer⁸ and others is the heterojunction model. In the heterojunction model the Cs-O layer is considered to be Cs_2O . A reduction in the work function results as the deposition layer thickness is increased, the bands bending until the normal work function of bulk Cs_2O is obtained. The heterojunction model is shown in Fig. 1b.

A pulsed Nd:YAG laser can be used to provide short (30 ps) pulses to excite electrons in the NEA devices. Gallium arsenide is a direct band gap material which, of the candidate materials, is the most readily available and well known. However, GaAs will not absorb the 1.06 μm wavelength radiation emitted by the laser; hence, frequency doubling is required. Other candidate materials are being considered which do not require the doubling of the 1.06 μm wavelength.

One of the great problems in using the existing literature to aid in the fabrication of NEA devices is the lack of characterization of the starting material, description of the fabrication steps, and the detailing of performance evaluation in conjunction with surface studies. The goal of the Los Alamos program is to develop a reliable material specification and NEA device fabrication process. To date we have used etch pit density measurements in conjunction with Auger Electron Spectroscopy and X-ray Photoelectron Spectroscopy to characterize the starting material and the surface layers respectively. Since the wafers must be annealed to about 600C, studies have also been initiated to find a suitable high temperature contact. We have also examined the use of anodic anodization for surface passivation.

Surface Defect Studies (EPD)

Diffusion lengths on the order of several microns are required by the model to correlate with the measured quantum efficiency. However, material of defect density less than $10^4/\text{cm}^2$ is desirable. This defect density appears to be much lower than would first be thought by a requirement of several micron diffusion lengths. A simple-minded calculation can be made relating the defect density to the amount of light that falls the farthest from the defect. If it is assumed that the electrons created in a circle will diffuse along a radius to the boundary of the circle and be captured, a weighted diffusion length can be calculated. The intensity weighted diffusion length is about one sixth the actual diameter. Thus, a diffusion length of 5 microns requires that the defects be about 30 microns apart, or a defect density of $\sim 10^{15}/\text{cm}^2$, which is what was observed making an etch pit density measurement. The best wafer was $\sim 10^{14}/\text{cm}^2$, again in accordance with the better 9% quantum efficiency measurement. As in situ activation of GaAs is a lengthy process, screening of the material to eliminate heavily defected wafers is necessary. In comparing wafers which could be activated successfully with those which could not, a correlation between etch pit density and performance was found.

Samples of gallium arsenide of (100) and (111) orientation, $10^{18}/\text{cm}^3$ to $10^{20}/\text{cm}^3$ zinc doped, and $10^2/\text{cm}^2$ to $10^4/\text{cm}^2$ etch pit density were obtained from a variety of manufacturers. Etch pits were studied using an ASTM-based procedure. Potassium hydroxide (KOH) at 400-450C was used to etch the surfaces. A Leitz metallurgical microscope and a Wilde M-5 were used to examine the surfaces at 125 x to 320 x. The results are summarized in Table 1. Figure 2 compares "good" and "bad" wafers. Fig. 3 shows the variation within a single lot from one manufacturer. The variation in EPD (over four orders of magnitude in the example shown) through a single lot demonstrates the need to screen each wafer as received.

Contact Development

Silver-manganese and nickel-silicon contacts were deposited onto GaAs samples. The resistivity as a function of temperature was continuously monitored from ~ 200 to 600C. The samples were examined before and after temperature cycling using Auger Depth Profiling. The silver-manganese couple has been used previously. This pair of materials has a wide miscibility gap that causes problems on heating. Gallium arsenide was coated with about 200nm of film deposited from a dual electron beam gun system. The films as deposited were uniform in composition before heating as seen in Fig. 4. After heating to 600°C, the silver and manganese phase separated, and the contacts did not appear to be stable with temperature. A contact made with silicon-nickel was suggested by Jackson and Kuan⁹. They proposed that the silicon would diffuse into the gallium arsenide making a p⁺ layer, ensuring a low contact resistance interface. Several stoichiometries were tried, with the Si_2Ni compound being the most successful. The nickel-rich silicides had a tendency to exhibit interdiffusion with the gallium arsenide, while the silicon-rich silicide did not. Auger depth profiles of the before and after heating of the Si_2Ni sample are shown in Fig. 5. Not

only did this sample show no significant change by Auger depth profiling, but it was the only one that did not show any visible changes after heating.

Surface Analysis Evaluation

Two surface analytical chambers were used to study the gallium arsenide. The first is a Physical Electronics Industries Model 545 scanning Auger microprobe with a spatial resolution of ~ 3 microns. The second system is a Leybold-Heraeus LH-10 hemispherical analyzer. An electron gun with a spot size ~ 100 microns was used for the Auger spectroscopy. Both systems also have X-Ray Photoelectron Spectroscopy capabilities. The electron energies were limited to 5 KeV or less with beam currents of 500 nA or less. These beam energies and fluxes should have minimized any potential damage to the sample and overlayers due to heating or electron stimulated desorption. Both systems were capable of ion beam sputtering. The base pressure in both systems was $<2.25 \times 10^{-10}$ Torr (3×10^{-8} Pa).

The GaAs was sputtered to remove surface oxygen and carbon before any activation was attempted. The surface composition was deficient in As as shown by Auger electron spectroscopy¹⁰. A heat treatment of $\sim 590^\circ\text{C}$ for 2-10 hours was found to be necessary to anneal out the sputter damage. The damage was estimated to be $\sim 2.5\text{nm}$ based on a Monte Carlo simulation¹¹. Prolonged heating on the order of a week was not found to be of any benefit. Since the sample was mounted under a tantalum cap, the step from the exposed area to the shielded area could be measured after removal from the system. Unless the material under the cap edge could escape into the vacuum, this step provides information about evaporation versus annealing at temperature. A sample that was heated for a week did not show any larger step than a sample that was sputtered and heated for shorter periods of time. The net step height was $\sim 150\text{nm}$ which is reasonable for the amount of ion sputtering used. Other research has shown that shorter annealing times are required to produce low energy electron diffraction patterns, but the limited coherence width of the electron beams limits the surface correlation to $\sim 100\text{nm}$ which is much less than the diffusion lengths required for good quantum efficiency. We believe that a good diffraction pattern is necessary for good photocathode performance, but is not sufficient since it is unable to "see" the limiting defects.

Once the sample was cleaned, activation was attempted. The cesium getter had been outgassed previously, and then the current increased so that the deposition rate would be ~ 1 layer/10 minutes. The photocurrent was monitored using an electrometer while biasing the cathode to ~ -90 volts. Until the work function dropped by several electron volts, no photoemission was observed. Then photoemission increased to a maximum, decreasing with further exposure. At this point the oxygen was admitted to the chamber until the emission reached a maximum and turned off. This process was repeated several times until the maximum attainable photoemission could be reached. This procedure is referred to as the "yo-yo" technique. The other method claimed to produce higher yields is the simultaneous cesium deposition and constant oxygen exposure to give a maximum rate of rise in the photocurrents. This procedure was tried and not found to be of any significant advantage. Another process that is recommended is the hi-low treatment. The substrate was heated to $\sim 600^\circ\text{C}$, cooled, and then activated. After the first activation, the substrate was heated to about 500°C , cooled, and then activated again. The second activation was supposed to increase the quantum efficiency, but again, we did not observe any benefit from the hi-low procedure except on a cathode surface that was not sputter cleaned. Unfortunately, no surface analytical equipment was present on this chamber. A spectrum of a fully activated cathode is shown in Fig. 6. The cesium and oxygen concentrations were very critical for high yields. If the cathode were further exposed to oxygen at 2.25×10^{-11} Torr (3×10^{-9} Pa), the cathode readily poisoned. Figure 7 shows a spectrum of a "dead" cathode. Clearly, high quantum efficiency can be maintained only in the cleanest of systems.

The work function measurements were taken by biasing the sample to -9v and measuring the cutoff of the secondary electrons. Figure 8 shows the change in the work function versus time. Assuming a constant sticking coefficient, the abscissa can be considered to be proportional to the overall coverage. No quantitative measurements were made to determine the amount of cesium present at the minimum of the curve. From other work¹¹, it may be inferred that the minimum occurs at about .5 monolayers. The overlayer structure has not been observed to be ordered as measured by low energy electron diffraction. Our work assumes little or no diffusion on the surface after the cesium arrives. If this is the case, then a Poisson distribution for the surface coverage may be valid. Using this growth model, a set of closed form solutions for the overlayer and substrate signal strengths may be computed. The results of these calculations¹² are shown in Fig. 9. The measured changes in signal strength agree well with the model, and allow a set of calibration curves to be developed for future cesiation.

ANODIC OXIDATION EVALUATION

The anodic oxidation procedure used by Sinclair¹⁴ and others is one in which the semiconductor is made the anode of an electrolytic cell¹⁵. The cathode is usually formed by a noble metal such as platinum. The electrolyte used here was phosphoric acid in water. A bias applied across the cell will create a field at

the semiconductor/electrolyte interface. While the constant bias configuration has been used by Sinclair, we also employed the constant current configuration. As the nonconductive oxide grows, its resistance will increase. In order to maintain a constant current, the bias must increase. If a constant bias is used, the current will drop. The oxide formed on the semiconductor is frequently soluble in the electrolyte; hence, dissolution is a competing process with oxide growth, particularly if the electrolyte has a low pH. Anodic oxidation has been considered useful in cleaning, protecting, and preparing the surface (after stripping) for activation. However, greater handling is required and the surface morphology is at risk.

Another cathode was prepared from the wafer that provided the 9% cathode. A four way preparation was tried by cleaning the cathode, etching* half the surface, rotating the sample by 90 degrees, and then anodizing only half the sample. Thus, four areas emerge in different states of preparation: 1) cleaned; 2) cleaned and etched; 3) cleaned and anodized; and 4) cleaned, etched, and anodized. The sample was stripped using ammonium hydroxide and distilled water. It was introduced to the ultrahigh vacuum system, taken through the procedures previously described, and activated. No significant difference could be found across any of the differently prepared sections. What was found to be critical was the removal of all the oxides and any surface carbon. However, some residual carbon has not been found to significantly reduce the performance of the cathodes. The net quantum efficiency was about 3-5% and reasonably uniform over the surface.

RESULTS

To date a number of significant properties relevant to the successful activation of a cathode has been found. The etch pit density seems to correlate with the observed maximum quantum efficiencies, i.e. high density gives low quantum yields, and low density gives high yields probably through longer diffusion lengths. The low value of the defect density required for high efficiencies is most likely related to the weighted diffusion length based on where the photoelectrons are created.

An ohmic contact of nickel-silicon that is stable at high temperature has been tried. This contact material is much more stable than the silver-manganese.

The cathode lifetime in the chamber is related to the quality of the vacuum for our experiments. Residual gas contamination would mask any cesium evaporation effects in this experiment even though the base pressure was 5×10^{-11} Torr (6.65×10^{-9} Pa). After cathode failure, an excess of oxygen was found on the cathode surface without a measurable change in the cesium concentration.

After any ion sputtering is used to remove carbon and oxides, the cathodes had to be annealed in order to obtain full quantum efficiency. This observation tends to confirm the importance of the near surface electrical properties of the cathode.

The layer of the cesium-oxygen used to lower the work function does not appear to be ordered. Previous workers using low energy electron diffraction did not observe any ordered patterns on the (100) surface of the gallium arsenide¹⁶. The Auger calculation, which assumes no ordering, describes the functional form of the data very well, lending further evidence for the lack of surface ordering.

Anodization does not appear to provide any benefit to the preparation process when sputter cleaning is available. The surface analytical techniques combined with the ion sputtering allow the preparation of an atomically clean surface for activation.

SUMMARY

The quality of the gallium arsenide substrate plays a direct role in the maximum quantum efficiency obtainable for a given cathode. Cesium and oxygen can reduce the work function on any p-type gallium arsenide crystal, but the quantum efficiency will be limited by the diffusion length of the material. The silicon-nickel contact material appears to be quite useful for high temperature cycling applications where annealing is required to remove damage due to ion sputtering. No significant interdiffusion was observed for this couple after temperature cycling to 600°C. Lastly, the cesium and oxygen layers are probably not ordered on the surface as evidenced by the agreement with the Auger intensity measurements and their correlation with a Poisson distribution for the surface coverage.

* Using $4 \text{ H}_2\text{SO}_4 : \text{H}_2\text{O}_2 : \text{H}_2\text{O}$.

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Table 1

A COMPARISON OF EPD AND PERFORMANCE

SAMPLE	MANUFACTURER	EPD MEAS/REPORTED(cm^2) DOPING LEVEL($/\text{cm}^3$)			RESULTS
300-1	Sumitomo	$<10^4$	10^2	$1.9-2.2 \times 10^{19}$	QE Wafer(RWS)
300-2	"	10^5-10^6			
300-3	"	$\sim 10^6$			
300-4	"	$\sim 10^6$			
300-5	"	$\sim 10^6$			
300-6	"	10^5-10^6			
300-7	"	$\sim 10^6$			
Sinclair Metal Specialties		10^3		6×10^{18}	$\sim 9\% \text{QE}$
"	Sumitomo	$>10^6$	$10^3?$	?	1%
100	Atomergic	10^5	10^4	9.5×10^{18}	1st Activated, $\sim 2\%$
200-3-1	Crystal Specialties	10^8	10^4	7.5×10^{18}	Could not Activate
Sinclair	ITT(EPI-CAAS)	$5 \times 10^3-10^6$	?	?	$\sim 6\%$

Figure 1. Dipole and Heterojunction Physical Models for Cs-O Photocathode

ATOMERGIC GaAs, (100), P, Zn, C.C. 9.5×10^{18} cc

CRYSTAL SPECIALTIES GaAs, (100), P, Zn C.C. 7.5×10^{18} cc

METAL SPECIALTIES GaAs, (100), 6×10^{18} cc Zn

SUMITOMO (100)

COMPARISON BETWEEN EPD AND ACTIVATION SUCCESS

Figure 2. A comparison between the EPD and activation success is shown. The Atomergic wafer ($EPD = 10^5/cm^2$) could be weakly activated to 2%. The Crystal Specialties ($EPD = 10^6/cm^2$) and Sumitomo ($EPD = 10^6/cm^2$) wafers could not be activated. The Metal Specialties wafer ($EPD = 10^3/cm^2$) produced 9% QE.

Figure 3. The range of EPD within a lot for one manufacturer is shown. SUMITOMO GaAs (HB) (100), P, Zn C.C.1.8--2.2E19cc Res. 4.2-5E-03 ohm-cm. Mob. 6.6--7E01 sq. cm./V.S. EPD 2E03 sq. cm.

Figure 4.

The Auger depth profiles of the silver-manganese contacts are shown. Fig. 4a is of the as-deposited film. The silver has a slightly higher concentration at the surface. This is probably due to a lack of stability in the source during the deposition. Fig. 4b illustrates the phase separation of the silver and manganese on heating. This observation is consistent with the binary phase diagram.

Figure 5.

The Auger depth profiles of the nickel-silicon contacts are shown. The as-deposited films (Fig. 5a) are very uniform and close to the design stoichiometry of NiSi. After heating (Fig. 5b) there was no visible change on the surface of the sample, and the depth profile is essentially the same. There was no observed interdiffusion with the gallium as observed on the silver-manganese contacts.

An Auger spectrum of the failed cathode is shown. This cathode had lost its quantum efficiency, and a re-activation was attempted without first "erasing" the previous layer. As can be seen, fairly small changes in this surface layer lead to a dead cathode. Probably one additional layer of cesium and about 25% more oxygen are enough to reduce the quantum efficiency to less than .5%.

Figure 8.

The work function versus coverage curve is shown. This curve was obtained by measuring the cutoff in the secondary electron distribution of the cathode surface while the cesium was being deposited. The shape and magnitude of this curve is in excellent agreement with previous measurements by other workers.

An Auger spectrum of the fully activated cathode surface is shown. The quantum efficiency of this cathode was ~6% for the He-Ne laser light just before the spectrum was taken. Note that the gallium and arsenic peaks are still easily measured, indicating a film thickness <3 atomic layers.

Figure 9.

The change in the Auger signal strengths versus coverage is shown. The top two curves are for the gallium and arsenic KLL transitions. The increasing curve is the signal strength for the cesium overlayer. The calculation has some very simple assumptions about the signal strengths and includes a Poisson distribution for the growth. This non-ordered approach for the overlayer growth is in agreement with the lack of the observed LFED patterns by other workers.