Electrostatic Deflection Binary Alloy Evaporator*

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A new technique for producing thin film binary alloys is discussed which used periodic deflection of an electron beam between two sources in a vacuum evaporator. This technique makes it possible to produce high quality binary alloys of predetermined ratios and with uniform composition throughout the thickness, as shown by x-ray diffraction analysis.

INTRODUCTION

THE study of electronic and optical properties of binary alloys requires films of the alloys having uniform composition and clean smooth optical surfaces.[1] To accomplish this, it is desirable to evaporate both constituents simultaneously in high vacuum. The integral mixing of the components during deposition reduces or eliminates the need for subsequent annealing which degrades the surface of the film. Various methods[2–5] have been applied in the past to produce binary alloys of particular materials with differing vapor pressures.

We have constructed an evaporation system which utilizes periodic electrostatic deflection of an electron beam to heat independently the two constituents of the alloy. This system satisfies the above requirements for the production of alloy films and can be applied to a wide variety of materials. The electron beam is directed alternately at each source and the amount of power delivered to each source is determined by a feedback arrangement from quartz crystal deposition monitors. The two monitors are well collimated so that each sees only one source.

I. ADVANTAGES AND DISADVANTAGES

The main advantage of our electrostatic deflection system is that it is possible to produce binary alloy films of a predetermined composition, and that the composition of the films is uniform throughout the thickness. In addition the technique can be used to produce virtually any binary alloy system. The uniform composition is possible because our electrostatic deflection systems can respond very quickly to maintain a constant ratio of evaporating materials. We have achieved switching times (the time it takes the electron beam to be switched from one source to the other) of 0.2 msec which we have found to be satisfactory. Our feedback system limits the system response time to about 50 msec. However this is not a disadvantage since 50 msec is still short compared with the thermal time constant of the sources.

A fast switching time is desirable to keep the heating of the sources uniform and avoid heating material which the beam strikes as it passes between the sources. By taking the high voltage for the switching circuitry from the electron gun high voltage supply, the angle of deflection is made independent of variation in the electron gun high voltage.

A disadvantage of our technique is that high voltage circuitry must be used and some problems of arcing have been encountered when using high vapor pressure materials.

II. APPARATUS DESCRIPTION

A block diagram of the complete system is shown in Fig. 1. An alloy of uniform composition is made by controlling the beam duty cycle which is defined as the ratio of time the beam spends on one boat to the time it spends on the other. This is accomplished by varying the frequency of the beam deflection from one boat to the other. The time that the beam remains undeflected per cycle is fixed, and by necessity is less than the period of the switching.

Material which is evaporated from each boat is independently monitored by quartz crystal film thickness monitors. Each monitor detects both the amount and rate of one of the components being deposited and provides voltages proportional to the amount and rate. These voltages are used to maintain a constant ratio of evaporated material in the following way.

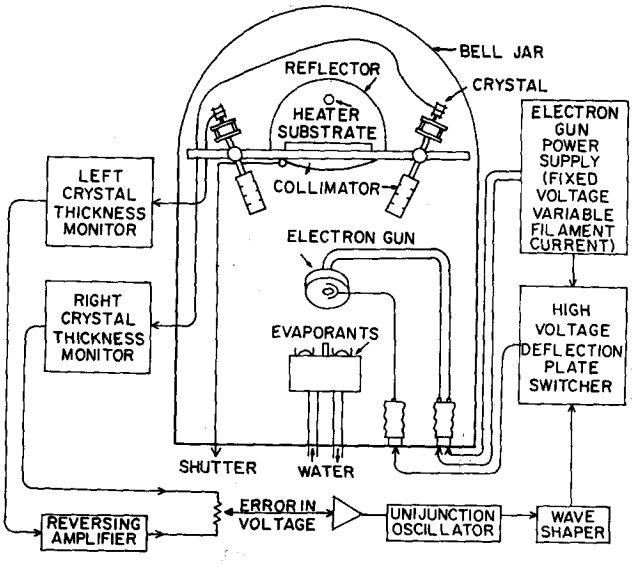

FIG. 1. Schematic drawing of the evaporation apparatus.

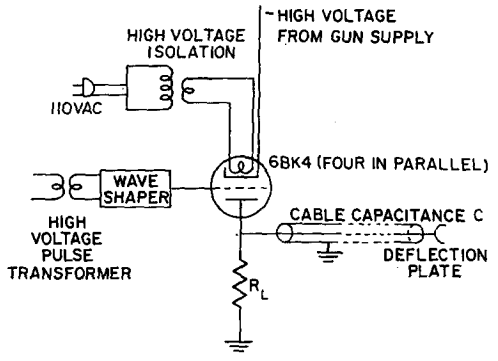

FIG. 2. Schematic drawing of the high voltage switching circuit.

The output voltage from one of the film monitors is reversed in sign by an inverting amplifier and added to the voltage of the other film monitor by a summing amplifier. The amplifier provides a negative voltage and serves to isolate the two monitors. The summing amplifier uses a 10-turn potentiometer at the input which is set to fix the ratio of alloyed materials. During an evaporation, when a steady state condition has been established which corresponds to the setting of this potentiometer, the output of the summing amplifier is zero. If a deviation should occur, an error voltage causes a change in frequency of the voltage controlled oscillator. This change of frequency alters the duty cycle of the pulse generator which controls the switching of the beam, and thus restores equilibrium. During an evaporation of an alloy, the change in frequency, Δf, is much less than f (i.e., $\Delta f/f \ll 1$), indicating that the system deviates only slightly from equilibrium. The voltages corresponding to either the total mass or the rate can be used for control.

With the exception of the electron gun, power supply, and high vacuum system, the entire system was constructed in our laboratory. The film thickness monitors are similar to commercially available units and utilize highly stable FET variable frequency reference oscillators. The voltage controlled oscillator is a unijunction-relaxation type, and it provides pulses at a frequency $f=f_0+\Delta f(V)$, where f_0 is a fixed frequency and $\Delta f(V)$ is the change in frequency caused by the error voltage V. These pulses are wave shaped by a one-shot multivibrator which provides a train of identical rectangular pulses of width t_1 and spacing $t_2=1/f$. These pulses are applied to the high voltage switch which deflects the electron beam between the two boats. The high voltage switch is shown in Fig. 2. The grid and cathodes of the tubes must be operated at high voltage since negative voltages are required for deflecting the electron beam. High voltage isolation of the signal input to the tubes is obtained by using a pulse transformer. This transmits pulses to a second wave shaper which provides fast risetime rectangular pulses for the grids. The electron beam thus periodically spends time t_1 on one boat and t_2-t_1 on the other. Capacitance between the deflection electrode and ground limits the actual switching time of the beam to values greater than R_LC, which in our case is about 200 μsec. This could be significantly reduced by replacing R_L by some 6BK4's operating in tandem with the other tubes and by reducing deflection plate leadwire and feed-through capacitance. However, 200 μsec switching time has been found to be adequate.

III. OPERATION

Alloys involving the evaporation of Ag, Au, Cd, Mg, Al, Sn, and Cr have been made, although α-phase Ag–Cd and Ag–Mg alloys have been most extensively studied. Accommodation of widely varying thermal time constants and evaporation power requirements for different metals is made by varying the setting of the pulse width. The maximum allowable deflection frequency is inversely proportional to the deflection pulse width.

Alloy composition is easily calculated if the crystal monitor calibration and summing potentiometer setting are known. It is found that the composition is repeatable for a given potentiometer setting if the crystal monitor collimators are not moved. The composition is checked by x-ray diffraction measurements of the alloy lattice constants. These measurements show that the composition is uniform to better than 1 at.% of the solute content throughout the thickness of the film. Samples are usually evaporated onto heated substrates in order to increase crystallite size for proper x-ray diffraction analysis. After the evaporation period, the substrates are immediately cooled to avoid degradation of the surface by residual gases. The evaporator is equipped with a shutter which shields the substrates from the boats. When a steady state rate of evaporation is established (indicated by a steady frequency of switching the beam), with the desired ratio (determined by the potentiometer setting), the shutter is opened for the evaporation period which is typically 100 sec, and then closed before the electron beam is turned off.

For evaporants such as cadmium which tend to scatter easily, it is necessary to maintain the evaporation rate below about 10 Å of alloy thickness per second at the substrate distance of 40 cm. Otherwise undesirable concentration gradients were produced along a line parallel to the line joining the two vapor sources. This gradient could be as much as 2.5% of the alloy solute content per centimeter along that line. This is in excess of the gradient expected because of geometric effects. Collisions between the evaporated atoms from the two sources are responsible for the effect. Essentially the beams of atoms sweep each other away from the center. Increasing the mean free paths by reducing the evaporation rate helps alleviate the problem.

Response time considerations are as follows: Feedback circuit response time and switching waveform period should be short compared to evaporant thermal time constant; the

latter should be short compared to total evaporation time; and the switching risetime should be short compared to switching waveform period, as previously discussed. Since the evaporant thermal time constant was long compared to the response time of the crystal monitors, it could be estimated by observing the decay of the evaporation rate after switching the electron beam away from the evaporant. The time constant thus obtained was on the order of 1 sec. Evaporants were placed in refractory metal inserts set in dimples in the cooled copper hearth. This was necessary because the electron gun was limited to rather low beam powers as a result of gun insulator problems. If more beam power were available, much shorter time constants could be obtained by removing the inserts.

A simple argument shows that uniform samples can be produced even if the thermal time constants are very short, provided the conditions in the last paragraph are satisfied. Assuming typical evaporation parameters: evaporation time of 100 sec, film thickness of 2000 Å, and a minimum switching frequency of 10 Hz; then the film thickness deposited per switching period is at most 2 Å, even assuming zero thermal time constant. Since this is less than a monolayer of any metal, a homogeneous film results. Therefore any thermal time constant consistent with the stability and responsivity conditions discussed is satisfactory.

The above design utilizes a straight-on Pierce type electron gun (Veeco Veb-6), but could be adapted to 180 or 270° deflection guns.

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