

Contra Costa College – Lawrence Hall of Science collaboration.
Spring 2013
Interdisciplinary Study of silver nanoparticles filtration.
ENGIN-230 Introduction to Circuit Analysis (ref 1)

As a practical application this class will include the study of a Scanning Electron Microscope (SEM)EM, and its use to analyze nanoparticles of silver. The silver nanoparticles are used in a filter paper, which can purify water of bacteria. This application is part of an interdisciplinary study with chemistry, biology and materials engineering.

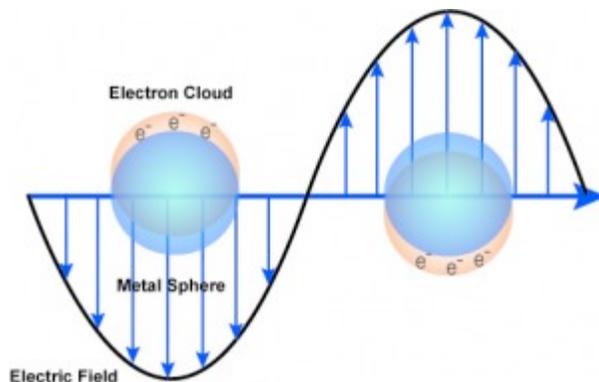
Lecture:

1/ Why nano-materials have different properties than bulk materials.

Nano particles have many different properties as illustrated by their interaction with photons. <http://nanocomposix.com/kb/general/plasmonics>

Plasmonic Nanoparticles

Plasmonic nanoparticles are metal nanoparticles (typically gold and silver particles with diameters ranging from 10-150 nm) that are highly efficient at absorbing and scattering light. By changing the size, shape, and surface coating, the color of the nanoparticles can be tuned across the visible and near-infrared region of the electromagnetic spectrum. Solutions of spherical gold nanoparticles are ruby red in color due to the strong scattering and absorption in the green region of the spectrum. Solutions of silver nanoparticles are yellow due to the plasmon resonance in the blue region of the spectrum (red and green light is unaffected).



Schematic of surface plasmon resonance where the free conduction electrons in the metal nanoparticle are driven into oscillation due to strong coupling with incident light.

The reason for the unique spectral response of silver and gold nanoparticles is that specific wavelengths of light can drive the conduction electrons in the metal to collectively oscillate, a phenomenon known as a surface plasmon resonance (SPR).

When these resonances are excited, absorption and scattering intensities can be up to 40x higher than identically sized particles that are not plasmonic. The brightness and tunability of plasmonic nanoparticle optical properties make them highly useful in numerous applications including molecular detection, solar energy materials, and cancer detection and treatment.

Plasmonic Nanoparticle Optical Properties

The vibrant colors of plasmonic nanoparticles occur because the conduction electrons on the surface of each nanoparticle vibrate when excited by light at a specific wavelength. These vibrations result in extremely bright colors that can be tuned by changing the particle size and shape (Figure 1). For a brief tutorial describing this effect, and how nanoparticle colors can be tuned, please refer to our [Plasmonics Knowledge Base article](#).

COLOR AT THE NANOSCALE

Plasmonic nanoparticles with absorption and scattering properties tuned for your application

[LEARN MORE >](#)



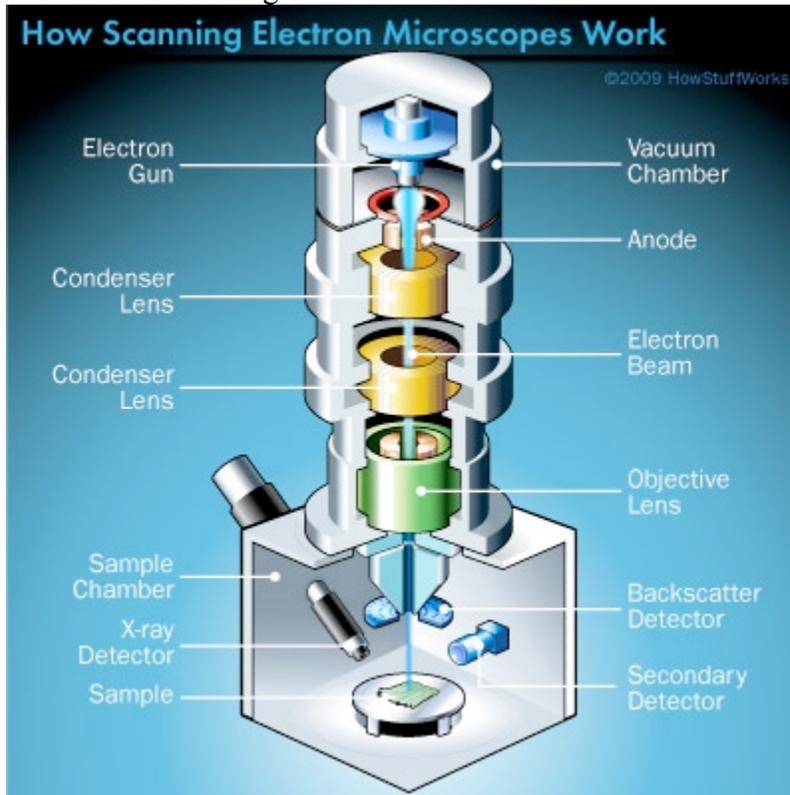
This is already spawning many companies, with a growing employment:

<http://www.nanotech-now.com/business.htm>
listed 540 companies in Dec 2010

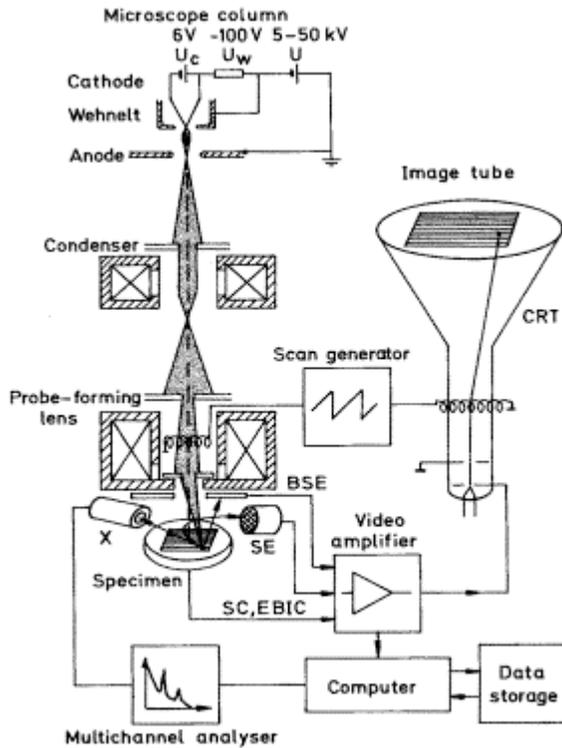
2/ Why the SEM is suited to the analysis of nano-materials.

The SEM can be used with EDS to identify the elements being observed. See Appx A. The SEM uses an electron beam instead of a light beam to inspect the object. The electrons hit the object, and some are “reflected” like light. These “backscattered” electrons are measured for each point on the object and displayed as an image on a computer screen. The magnification can be much greater than for light because the electron has a shorter wavelength than visible light.

2A/Basic functioning of the SEM.



The picture shows the main components of a typical SEM



The electrons are generated as in a cathode ray tube and the circuit design is similar. The anode accelerates the electrons toward the target – in a Sem this is often a variable – e.g 5 kvolts or 15 kvolts – these can give different images. A CRT is a sealed tube, but the SEM must allow for samples/targets to be introduced, and so it has a vacuum pump.

The SEM used in this lab is:

HITACHI TM3000 SEM Characteristics

- Accelerating voltage: 5 or 15 kV, tungsten source
- Magnification: X 15 to X 30,000
- 30 nm resolution
- Backscattered detector only
- Charge reduction mode (which uses higher chamber pressure) allows imaging of uncoated samples
- Easy to use and portable

This microscope joined the lab in 2010 and is used for demos, quick examination of genetic mutants, student projects and a myriad of samples needing a quick "look see".

In the SEM and electron microprobe, the sample is bombarded by a focused beam of electrons. Most incident electrons, rather than penetrating the sample in a linear fashion,

interact with specimen atoms and are scattered. They follow complicated twisting paths through the sample material, losing energy as they interact. The scattering events are of two types either elastic or inelastic. In elastic scattering, the electron trajectory changes, but its kinetic energy and velocity remain essentially constant. This is because of the large difference between the mass of the electron and atomic nuclei. Alternatively, in inelastic scattering, the trajectory of the incident electron may be only slightly perturbed, but energy is lost through interaction with the orbital electrons of the atoms in the specimen. Inelastic interactions produce a number of effects:

- Secondary electrons (SE)
- Backscattered electrons (BSE)
- Cathodoluminescence (CL)
- Continuum x-ray radiation (bremsstrahlung)
- Characteristic x-ray radiation
- Phonons

See appendix B for a description of the scanning circuits, and the generation of the high voltage - **Understanding The TV Horizontal Output Stage**

LABORATORY EXERCISE:

Use of SEM to characterize the nanoparticles of silver in the filter paper.

The filter paper with nanoparticles of silver was prepared by the CCC chemistry Dept.

This was then mounted on an aluminum stub using a carbon filled adhesive to ground the paper as well as possible.

There were 10 students in the class, and each one was able to take turns at operating the SEM, and to learn to search for the silver – by first having a low magnification and then zeroing in on a chosen particle using greater magnification.

They learned that the SEM is limited to examining the surface of the sample, and so nanoparticles of silver in the crevasses of the paper would not be seen.

We were able to identify bright spots (that is – spots that reflected many electrons) on the paper – and had to assume that was silver (since there was no EDS.)

On amplifying the bright spots, it was seen that they were typically about 3 microns or 3000 nanometers. This is probably because the nanoparticles tend to aggregate into clumps. Also the SEM cannot easily resolve submicron images because of vibration and other factors (it is a portable instrument).

The electron cloud due to the paper being a dielectric also contributed to the resolution. The students were able to see how that was affected by changing the electron energy,

and also going to the “Charge up reduction mode” – this used a partial vacuum to allow ions which neutralize the unwanted electrons.

The students hands on experience gave them an appreciation of the power of the SEM, and also of the skill of the operator in interpreting the images.

The students wrote a comparison of the SEM used with a TEM (transmission electron microscope).

References

1. ENGIN-230 Introduction to Circuit Analysis

4 Units: 54 hours of lecture, 54 hours of laboratory, per term, LR, DG, CSU, UC

Co-requisite: MATH 290 and PHYS 230

This course provides primary emphasis on developing models and techniques for analyzing the natural and forced response of passive linear networks. Models and circuit applications for diodes, transistors, and operational amplifiers are introduced. Laboratory experiments with each of the fundamental circuit types and related test equipment are conducted. Not repeatable.

Appendix A

http://www.charfac.umn.edu/instruments/eds_on_sem_primer.pdf

Energy Dispersive Spectroscopy on the SEM:

A Primer Bob Hafner

This primer is intended as background for the “EDS Analysis on the SEM” course offered by the University of Minnesota’s Characterization Facility. You must learn this material prior to the hands-on training sessions. It is also assumed that you have a working familiarity with the content in the

“Scanning Electron Microscopy Primer”. Good sources for further information are:

“Scanning Electron

Microscopy and X-Ray Microanalysis” by Joseph Goldstein et al; and, “X-ray and Image Analysis in

Electron Microscopy” by John J. Friel.

The Big Picture

Backscattered electron images in the SEM display compositional contrast that results from different

atomic number elements and their distribution. Energy Dispersive Spectroscopy (EDS) allows one to

identify what those particular elements are and their relative proportions (Atomic % for example).

Initial EDS analysis usually involves the generation of an X-ray spectrum from the entire scan area of the SEM. Below is a secondary electron image of a polished geological specimen and the corresponding X-ray spectra that was generated from the entire scan area. The Y-axis shows the counts (number of Xrays received and processed by the detector) and the X-axis shows the energy level of those counts. The EDS software we have, “Noran System Six” (NSS), is quite good at associating the energy level of the X-rays with the elements and shell levels that generated them.

Appendix B



Understanding The TV Horizontal Output Stage

One of the biggest mysteries in television receivers and video monitors is the operation of the horizontal output stage. The basic concept of this stage has not changed for many years, but scan derived supplies and startup and shutdown circuits have made the horizontal output stage one of the most feared circuits in television receivers and NTSC video monitors.

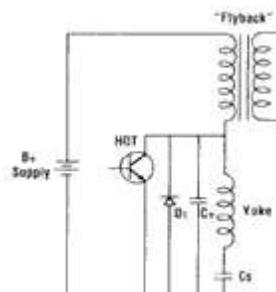


Fig. 1: Simplified horizontal output stage.

Many servicers have difficulty relating horizontal output symptoms to the possible cause, and often misinterpret circuit voltages and other troubleshooting clues. Many of these difficulties arise because servicers don't have a good, practical understanding of how the horizontal output stage functions. This Tech Tip explains the operation of the horizontal output stage in terms of the key

components, currents and voltages.

Key Components

All horizontal output stages operate virtually the same regardless of make or model. All output stages drive a sawtooth current into the primary winding of the flyback transformer and receive power from the main B+ supply. The B+ supply can deliver peak currents of several amps while maintaining a regulated voltage of about 130 VDC. The peak-to-peak current required by the output stage depends on the CRT size, the number of scan derived supplies, and whether the chassis is color or B&W.

Figure 1 shows a simplified horizontal output stage. It consists of six key components: 1) horizontal output transistor (Q1); 2) flyback transformer (flyback); 3) retrace timing capacitor or "safety cap" (Ct); 4) damper diode (D1); 5) horizontal yoke; and 6) the yoke series capacitor (Cs). Let's take a closer look at the role that each of these components plays in the operation of the output stage.

Fig. 1: Simplified horizontal output stage.

Output Transistor

The horizontal output transistor (HOT) is a switch. It provides a path for current to flow through the flyback's primary winding and horizontal yoke as shown in Figure 2. The HOT is switched on and off by a signal applied to the base. Because this is a power transistor, a current drive signal is needed. This drive current is supplied by the horizontal driver and driver transformer. In addition to current step-up, the driver transformer provides Impedance matching.

The horizontal output transistor passes current levels ranging from 200 mA in a small B&W chassis to 1.5 amps in large screen color chassis that have multiple scan derived power supplies. These values are the average value and peak current may reach 4 amps.

If you remember your transistor theory you know that the collector current equals the base current multiplied by the current gain (beta). Therefore, the base drive current must be sufficient to produce the required collector current and may be as much 100-300mA. If the base drive is

insufficient the emitter-to-collector resistance of the conducting HOT will be too high and the transistor will get too hot. Sufficient drive is also important for fast switching.

The horizontal output transistor is switched on and off at the horizontal frequency of 15,734 Hz. The horizontal oscillator (which controls the driver stage) begins to turn the horizontal output transistor on approximately 30-35 μS before horizontal sync as shown in Figure

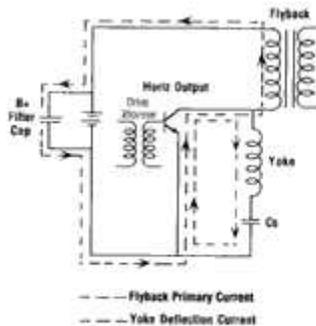


Fig. 2: Horizontal output transistor conduction current paths

3. The HOT conducts until the start of horizontal sync and then is abruptly turned off.

The time it takes to switch the HOT from on to off is important. As the transistor is switched, the emitter-to-collector resistance changes from <5 ohms (on) to >10 megohms (off). The current flowing through the transistor during the on/off transition produces heat, and, the longer the transition the greater the heat buildup and the greater the chance of thermal failure.

The drive current produces a voltage waveform at the base of the HOT that is

Fig. 2: Horizontal output transistor current conduction paths

similar to a squarewave. Schematics

Fig. 3: The horizontal output transistor is turned on 30-35 μsec. before horizontal sync and is

turned off at horizontal sync.

show peak-to-peak values ranging from 5 to 30 volts, but much of the amplitude is due to voltage spikes caused by the switching action. The waveform only confirms the presence of drive to the HOT. It cannot confirm if the base current drive is adequate for normal operation. Reduced

or improper drive results in transistor heating, reduced deflection (width), picture foldover, and shortened HOT life.

Flyback Transformer

The output transformer is called the flyback or IHVT. (An IHVT is a flyback transformer that includes the high voltage multiplier). The flyback is primarily responsible for developing high voltage. It

Fig. 4: The Flyback transformer contains one primary winding and several secondary windings.

is constructed with a powdered iron or ceramic core to work efficiently at high frequencies.

The flyback includes one primary winding and many secondary windings. The main secondary winding supplies voltage pulses to the voltage multiplier. Other secondary windings supply CRT filament power, keying pulses, and scan-derived power supplies, as shown in Figure 4.

The primary winding is in series with the HOT and B+ power supply, and is driven with pulses from the switching action of the HOT. The resulting switching pulses are typically 700 to 1100 VPP. To understand how these pulses are produced you need to recall some basic inductor theory.

The voltage induced across an inductance is given by the formula $V = L(di/dt)$, where L is the inductance of the coil and di/dt is the rate that the current through the coil changes. The current in the flyback primary raises at a linear rate when the HOT is conducting. This produces a constant amount of induced voltage in the flyback windings. But, when the HOT is abruptly turned off, the magnetic field in the flyback core rapidly collapses and induces a high voltage into the flyback's primary and secondary windings, as shown in Figure 5.

The rate that the magnetic field collapses is controlled with timing components. If it weren't, induced voltage spikes of several thousand volts would be produced across the flyback primary. These spikes would exceed the breakdown rating of the horizontal output transistor and flyback, and produce excessive high voltages to all flyback windings.

Retrace Timing Capacitor

The retrace timing capacitor slows down the rate of the flyback's collapsing magnetic field. This is a very critical function. If the retrace capacitor's value decreases, or if it opens, the amplitude of the flyback pulse will increase several thousand volts. To minimize this danger, several smaller value capacitors are usually connected in parallel with the retrace capacitor. Also, safety shut-down circuits are added to disable the output stage should the high voltage increase to unsafe levels. Because of the key role that the retrace capacitor plays in controlling the induced voltage and CRT high voltage it is often called the "safety capacitor".

Damper Diode

The damper diode completes the resonant current path for the flyback primary and deflection yoke by turning on during the time when the current through the HOT reverses. If the damper diode opens the HOT is forced to operate in reverse breakdown which will cause the transistor to fail. The damper is a fast switching, high current diode.

Horizontal Yoke

The rising and falling sawtooth current flowing in the yoke produces horizontal electron beam deflection. Because it is part of the output stage, the yoke also affects retrace timing.

Yoke Series Capacitor

The yoke series capacitor has four functions: 1) matching the resonant timing of the yoke current; 2) helps establish retrace time; 3) prevents a fixed DC bias on the yoke; 4) shapes the deflection current to match the CRT.

Understanding Output Stage Operation

Now let's put the components together and see how the whole circuit operates. We will analyze the output stage in two parts according to the major functions it performs: 1) Flyback primary current and retrace time, and 2) Horizontal deflection.

Fig. 5: The rapidly collapsing magnetic field of the flyback transformer when the horizontal output transistor is switched off produces a high voltage pulse.

The first function, flyback primary current

and retrace time, is responsible for producing the CRT high voltage, focus voltage and scan derived supplies. The second function, as its name implies deals with deflecting the electron beam.

Although these two functions interact, discussing them separately will help you to better understand the operation of the output.

Flyback Primary Current and Retrace Time

Figure 6 shows the flyback action and current paths at 4 times during one output cycle, beginning with the horizontal output transistor turning on. When the HOT is turned on current flows into the flyback's primary from the Bt power supply. All of the power needed by the output stage, including the secondaries, is delivered to the circuit from the B+ supply during this time. The current and magnetic field in the flyback's core continue to build until the transistor is turned off.

During the next 3 periods the magnetic energy expands and collapses in the flyback producing current which charges and discharges the retrace timing capacitor. The current flow through the flyback primary winding transfers power to the secondary and its loads.

The magnetic field that was stored in the flyback's core begins to collapse immediately after the HOT is turned off. This is the beginning of retrace time and corresponds with the start of horizontal sync. With the HOT switched off the retrace timing capacitor is effectively placed in parallel with the flyback primary.

Thus, a resonant circuit is formed as shown in Figures 6b & c. The time constant of the resonant circuit is determined mainly by the value of the retrace capacitor and the inductance of the primary winding. The yoke components in parallel with Ct, (the yoke and Cs) also have an affect on retrace timing.

The collapsing magnetic field causes current to flow through the low impedance of the Bt supply's filter capacitors and into Ct. This current charges Ct and produces the large pulse at the HOT collector.

After the magnetic field has completely collapsed, Ct begins discharging which

causes current flow back into the primary in the opposite direction. A magnetic field rebuilds in the opposite polarity. This action completes the 2nd part of retrace and corresponds to the falling portion of the HOT collector pulse. A properly operating output stages has a retrace time (flyback pulse duration) of 11.3 to 15.9 μ sec.

When C_t has completely discharged the magnetic field begins to collapse. The collapsing field induces a voltage with a polarity that forward biases the damper diode. The damper diode serves as a switch and allows the magnetic energy (current) in the flyback and yoke to decay at a controlled rate. (The damper diode would not be needed if the horizontal output transistor could conduct current in either polarity).

With the damper diode turned on the circuit is highly inductive, so the current in the flyback primary once again slowly increases. Approximately 18 μ S latter the horizontal output transistor is once again turned on and the cycle repeats.

The flyback transformer works like any other transformer in that the energy in the primary is transferred to the secondaries. If all of the secondary loads were open most of the energy stored in the magnetic field would return back to the primary circuit. But the secondary circuits draw power from the primary. Thus, as the load on the secondary windings increases, more current flows in the primary and more current is drawn from the B_t supply. Some problems, such as a shorted secondary load circuit or a shorted flyback winding, may cause such a great load that the circuit cannot compensate for the power demand. This causes the horizontal output transistor to overheat and short, the flyback primary to open, or the $B+$ supply to fail.

fig. 6: The alternating flyback current, the current waveform, and the flyback voltage pulse for

one complete horizontal cycle.

Horizontal Yoke Deflection

The second major function of the horizontal output stage is to provide deflection current. The HOT's collector current is split between the flyback and

the horizontal yoke. Both paths share the damper diode and retrace timing capacitor.

Capacitor C_s , which is in series with the yoke has four functions: 1) it is primarily responsible for determining time constant of the deflection portion of the horizontal output stage; 2) it has an affect on retrace time; 3) it prevents a fixed DC bias from developing on the yoke that would cause improper picture centering; 4), it shapes the sawtooth rise in deflection current to match the slight curvature of the CRT.

Figure 7 shows the yoke deflection current at the same times as Figure 6. When the HOT is turned on the bottom side of the yoke series capacitor, C_s , is connected to

Fig. 7: Deflection current and beam position for one complete horizontal cycle.

the top of the yoke. Because C_s is fully charged, it begins discharging through the horizontal output transistor. The resulting current produces an expanding magnetic field in the yoke which moves the electron beam from the center of the screen towards the right side.

When the horizontal output transistor opens, the retrace timing capacitor is added to the circuit, as shown in Figure 7b. This increases the resonant frequency and causes the yoke's magnetic field to rapidly collapse. This is the beginning of retrace during which the beam is snapped from the right side of the screen back to the center. The induced voltage causes current to flow and returns the energy that was stored in the yoke's magnetic field to capacitors C_t and C_s . The retrace timing capacitor is replenished with charging current from the flyback transformer and becomes the current source for the yoke current.

During the 2nd part of retrace C_t and C_s discharge and force the current to flow in the opposite direction, as shown in Figure 7c. The timing is identical to the first part of retrace and the beam moves quickly from the center to the left side of the screen.

When capacitors C_t and C_s are fully discharged, the yoke's magnetic field begins to collapse, as shown in figure 7d. The induced voltage forward biases the damper diode into conduction. (Notice

that this occurs at the same time as it did in Figure 6).

The circuit's timing is now determined by the yoke and capacitor C_s and agrees with the timing during the right trace time. The yoke's collapsing magnetic field produces current through the damper diode which returns energy to the circuit and charges C_t .

When the yoke's magnetic field is collapsed, the damper diode stops conducting. The horizontal output transistor must immediately begin to conduct, otherwise there will be horizontal non-linearities in the center of the raster.

To simplify our explanation of the horizontal output stage we analyzed the flyback and yoke function separately. But these circuits are not independent: the flyback current is transferred to the yoke by the retrace timing capacitor C_t and the yoke and flyback currents share the conduction time of the horizontal output transistor, damper diode and retrace timing capacitor. Because of this interaction, most problems in the horizontal output circuits affect both the flyback and yoke current