

INSTRUMENTATION OF ESR SPECTROSCOPY

1.KLYSTRONS

Klystron tube acts as the source of radiation. It is stabilized against temperature fluctuation by immersion in an oil bath or by forced air cooling. The frequency of the monochromatic radiation is determined by the voltage applied to klystron. It is kept a fixed frequency by an automatic control circuit and provides a power output of about 300 milli watts.

2. WAVE GUIDE OR WAVEMETER

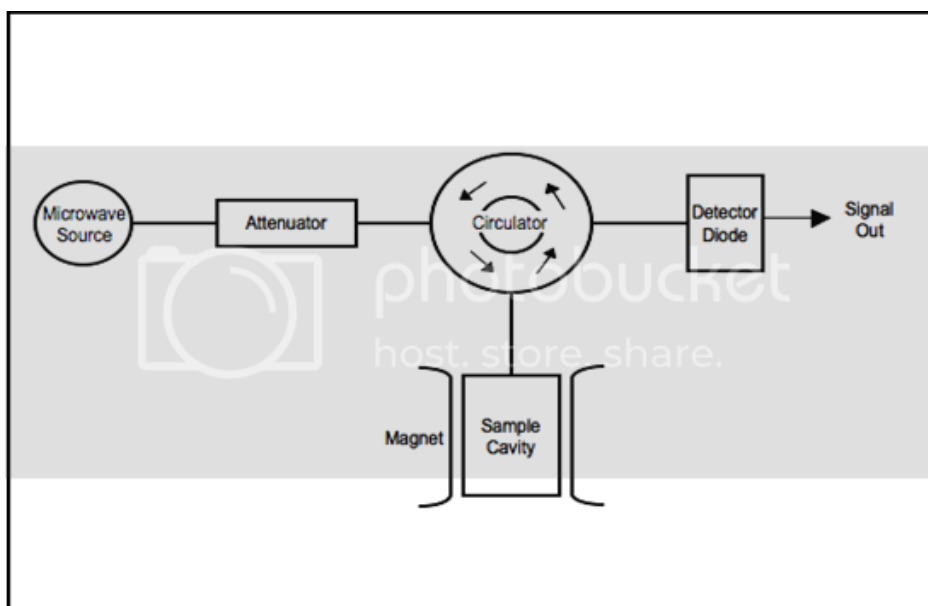
The wave meter is put in between the oscillator and attenuator to know the frequency of microwaves produced by klystron oscillator. The wave meter is usually calibrated in frequency unit (megahertz) instead of wavelength. Wave guide is a hollow, rectangular brass tube. It is used to convey the wave radiation to the sample and crystal.

3. ATTENUATORS

The power propagated down the wave guide may be continuously decreased by inserting a piece of resistive material into the wave guide. The piece is called variable attenuator and used in varying the power of the sample from the full power of klystron to one attenuated by a factor 100 or more.

4.ISOLATORS

It is a non-reciprocal device which minimizes vibrations in the frequency of microwaves produced by klystron oscillator. Isolators are used to prevent the reflection of microwave power back into the radiation source. It is a strip of ferrite material which allows micro waves in one direction only. It also is being stabilizing the frequency of the klystron.



Block diagram of a typical spectrometer

5. SAMPLE CAVITIES

The heart of the ESR spectrometer is the resonant cavity containing the sample. The sample is contained in a resonance cavity. Rectangular TE₁₂₀ cavity and cylindrical TE₀₁₁ cavity have widely been used. In most of the ESR spectrometers, dual sample cavities are generally used. This is done for simultaneous observation of a sample and a reference material. Since magnetic field interacts with the sample to cause spin resonance the sample is placed where the intensity of magnetic field is greatest. A measure of quality of the cavity is 'Q factor' which is defined as

The sensitivity of the spectrometer is directly proportional to this value of Q.

Rotable cavities and dual cavities have also been used respectively for study in anisotropic effect in single crystal and simultaneous spectroscopic observation of a sample and standard.

6. COUPLERS AND MATCHING SCREWS

The various components of the micro wave assembly to be coupled together by making use of irises or slots of various sizes.

7. CRYSTAL DETECTORS AND HOLDERS

A Silicon crystal detectors, which converts the radiation in D.C., has widely been used as a detector of microwave radiation. Microwave Bridge such as magic T and hybrid ring variety are most common.

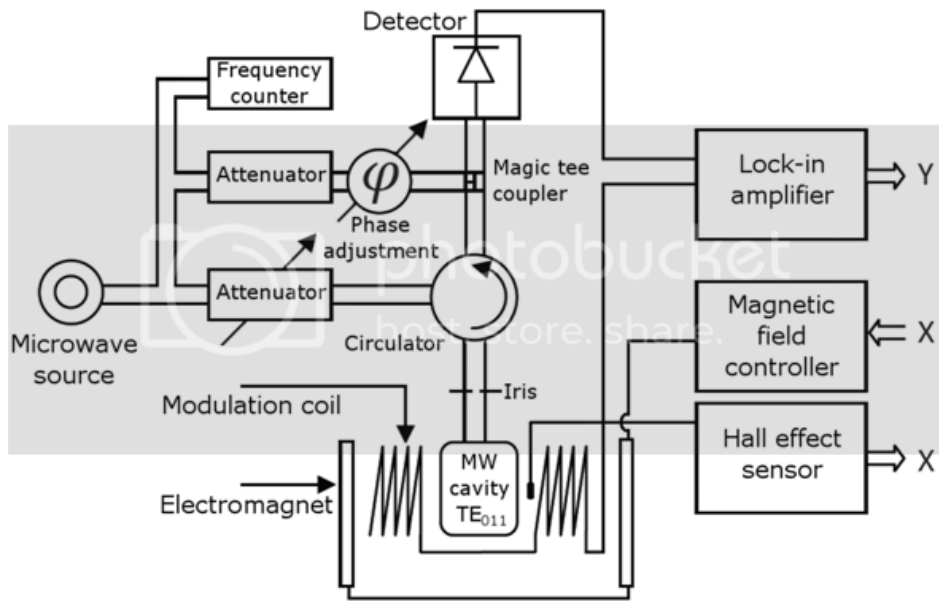
8. MAGNET SYSTEM

The resonant cavity is placed between the poles pieces of an electromagnet. An electro magnet capable of producing magnetic field of at least 5000 gauss is required for ESR. The field should be stable and uniform over the sample volume. The stability of field is achieved by energizing the magnet with a highly regulated power supply.

The ESR spectrum is recorded by slowly varying the magnetic field through the resonance condense by sweeping the current supplied to the magnet by the power supply. This sweep is usually accomplished by with a variable speed motor drive. Both the magnet as well as the power supply may require water cooling.

9. MODULATION COIL

The modulation of the signal at a frequency consistent with good signal noise ratio in the crystal detector is accomplished by a small alternating variation of the magnetic field. The variation is produced by supplying an A.C. signal to modulation coil oriented with respect the sample in the same direction as the magnetic field. If the modulation is of low frequency (400 cycles/sec or less), the coils can be mounted outside the cavity and even on the magnet pole pieces. For higher modulation frequencies, modulation coils must be mounted inside the resonant cavity or cavities constructed of a non-metallic material e.g., Quartz with a tin silvered plating, because metallic penetration is not very effective in case of higher modulation frequencies.



SCHEMATIC DIAGRAM OF AN ESR SPECTROMETER

10. DISPLAY DEVICES

In order to adjust the spectrometer and to observe the signal, a cathode ray oscilloscope is used along with the strip chart or X-Y recorder for recording the signal.