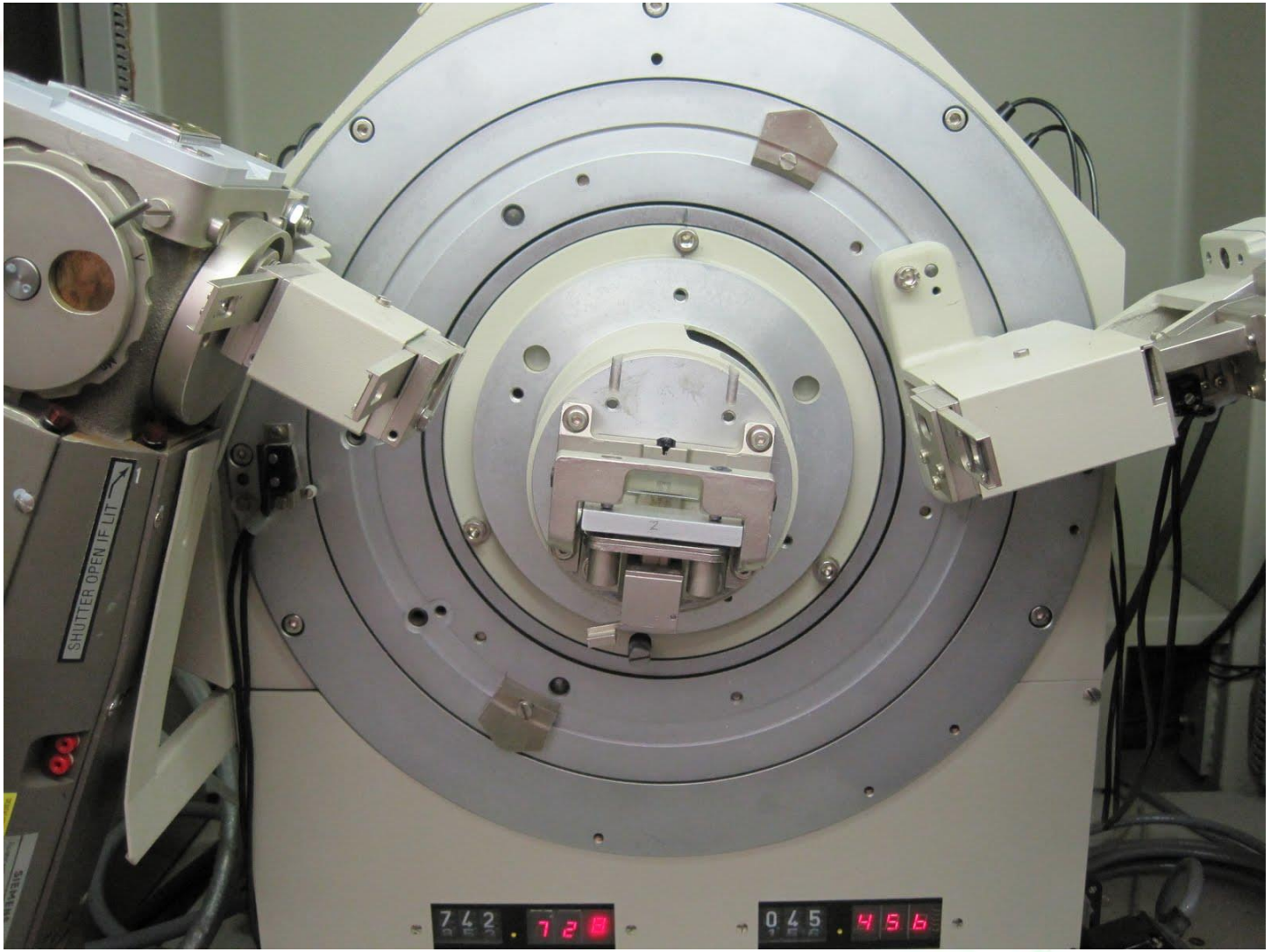
chem 5390

Advanced X-ray Analysis

LECTURE 9

Dr. Teresa D. Golden
University of North Texas
Department of Chemistry

Instrumentation



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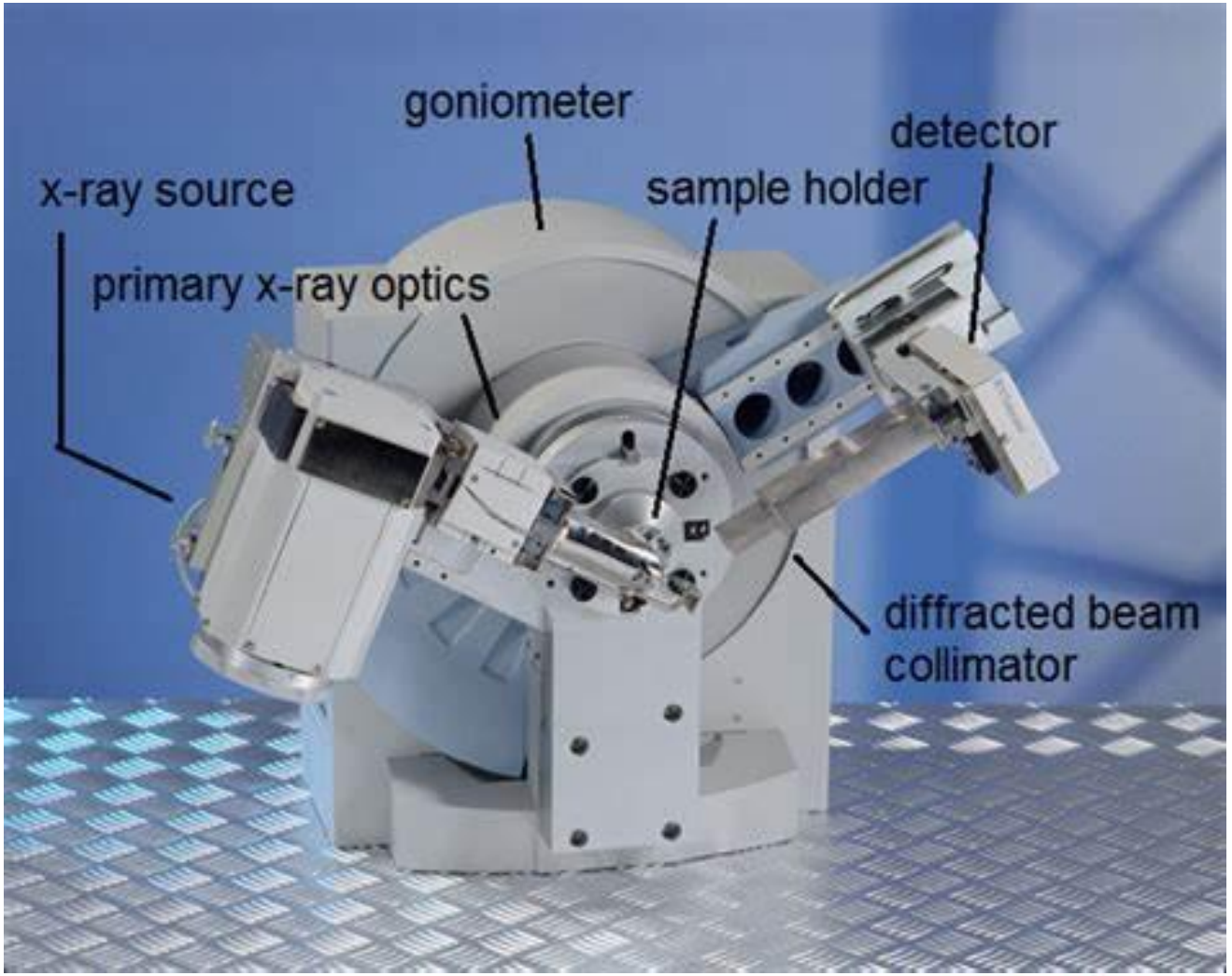
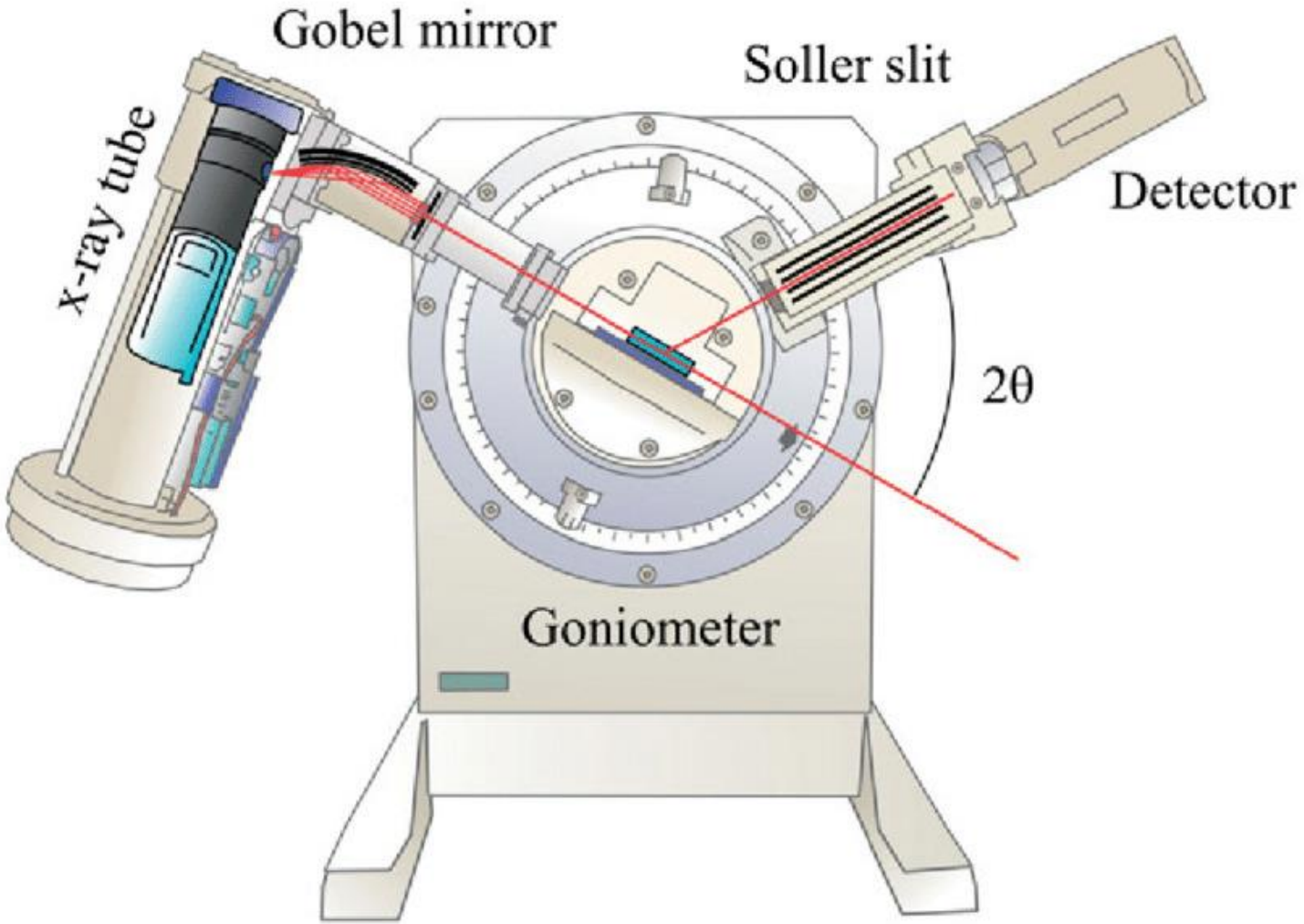
Instrumentation



Instrumentation



Instrumentation

A. X-ray Source Components

Components for the source include:
-Line voltage supply
-high-voltage generator
-x-ray tube

X-ray source requires
-high photon output
-high specific intensity
-selectable levels kV and mA
-stable output

Instrumentation

A. X-ray Source Components

1. Line-voltage supply

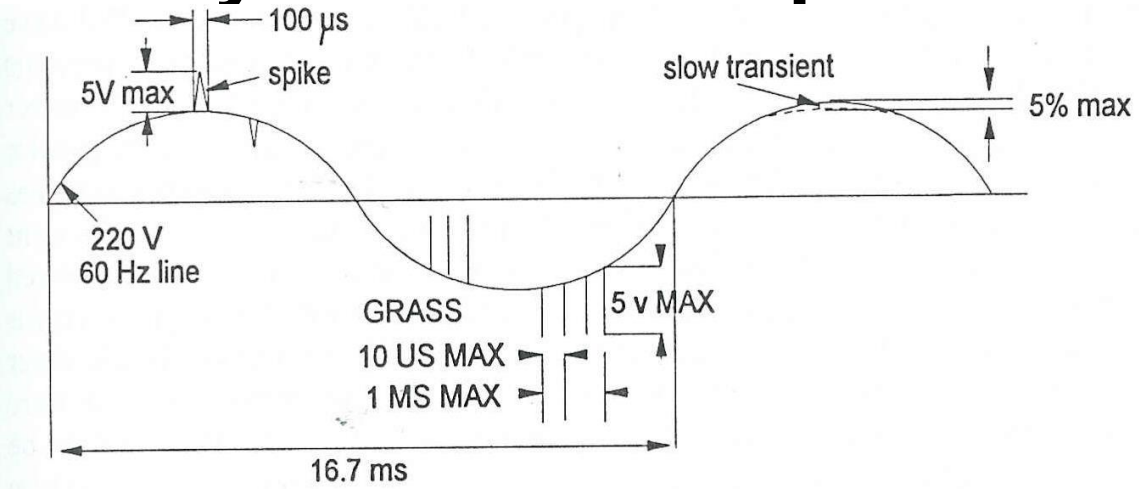
Usually 110 or 220 V

Variations in line voltage are due to:

- **a slow (mins or hrs) variation of voltage level**
- **cycle variation in the amplitude of the waveform**
- **superimposed short-term (msec) burst of high voltage spikes**

Instrumentation

A. X-ray Source Components



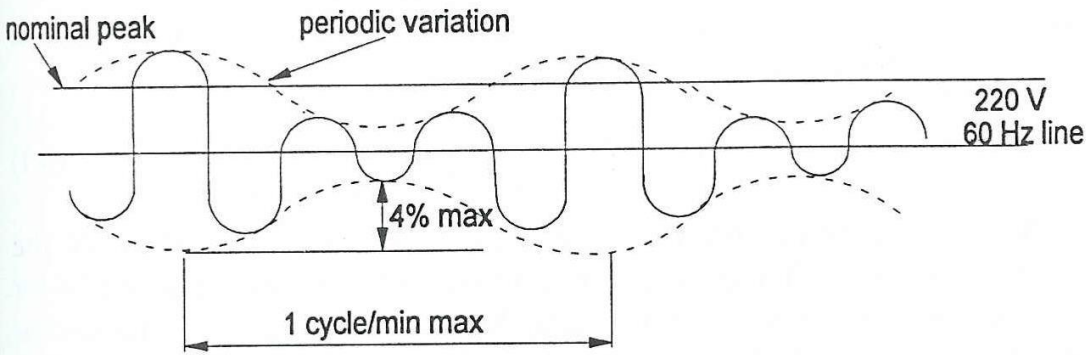


Figure 4.2. Waveshape aberrations for an ac source.

Instrumentation

A. X-ray Source Components

1. Line-voltage supply

The high-voltage generator can usually handle the first two variations (within ±10%), it is the last variation that can be a problem.

The generator contains stabilization circuits, but the response of these circuits is finite. For very short duration spikes, a burst of excess voltage will pass to the x-ray tube.

These spikes can give counting and display problems. Can use an in-line isolation transformer to clean the line-voltage supply.

Instrumentation

A. X-ray Source Components

2. High-voltage generator

Purpose - transforms line-voltage to supply 10,000 to 50,000 V in steps of 5000V.

Types of generators:

- **constant-potential**
- **half-wave rectified**
- **full-wave rectified**

Instrumentation

A. X-ray Source Components

2. High-voltage generator

Recently manufacturers have moved to high-frequency types, which have a lower cost, smaller size and weight, with greater conversion efficiencies (less heat loss).

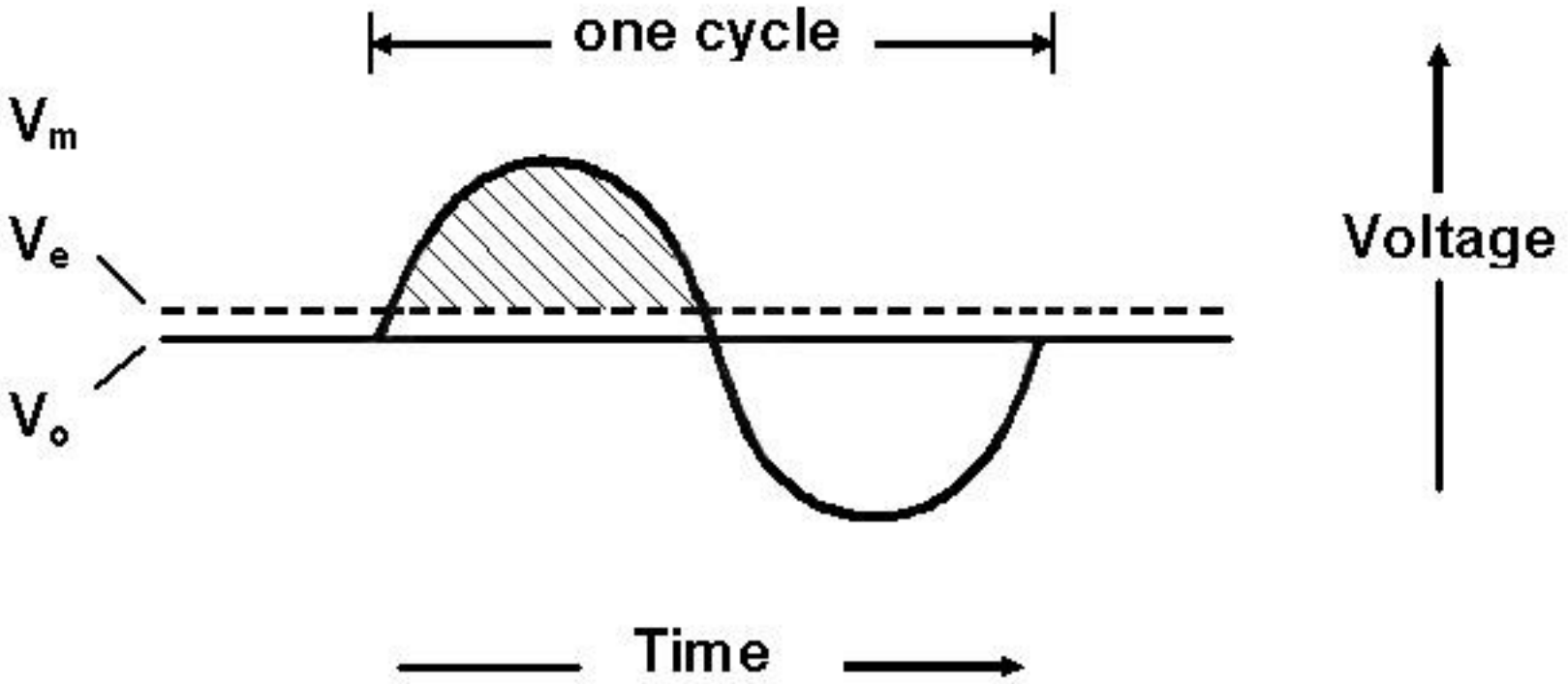
+/- 0.01% for a +/-10% variation in the line voltage and +/- 5°C change in ambient temperature.

Instrumentation

A. X-ray Source Components

2. High-voltage generator

- Half-wave generator



Instrumentation

A. X-ray Source Components

2. High-voltage generator

- Half-wave generator

Cycle starts at zero volts (V_o), reaches a maximum (V_m) at 1/4 of the cycle, drops to zero at 1/2 of the cycle, drops to a minimum ($-V_m$) at 3/4 of the cycle, and at a full cycle is back at zero.

Instrumentation

A. X-ray Source Components

2. High-voltage generator

- Half-wave generator

If the excitation potential for a characteristic line (e.g. CuKα) is V_e, the value of V_e is only exceeded for a specified period of the cycle. This effective part is called the <u>duty cycle</u> of the generator (~30% of the cycle).

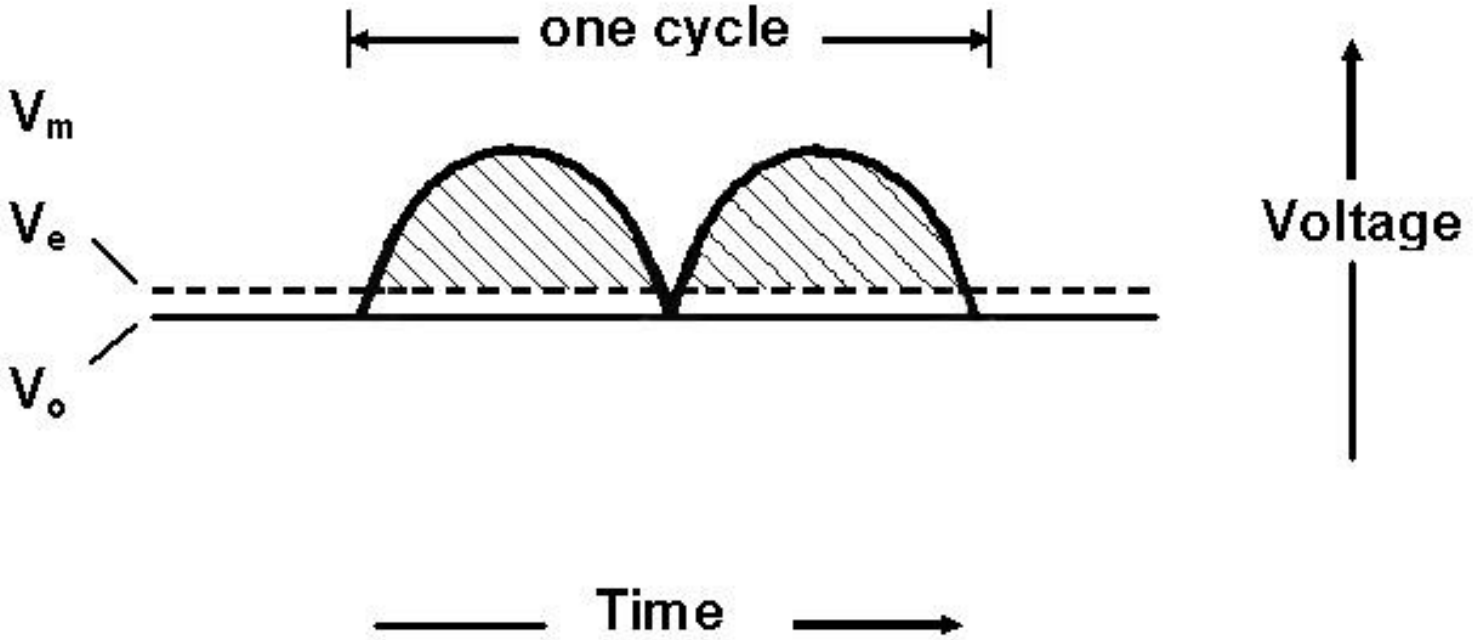
Instrumentation

A. X-ray Source Components

2. High-voltage generator

-Full-wave generator

If the line voltage is rectified get a doubling of the duty cycle.

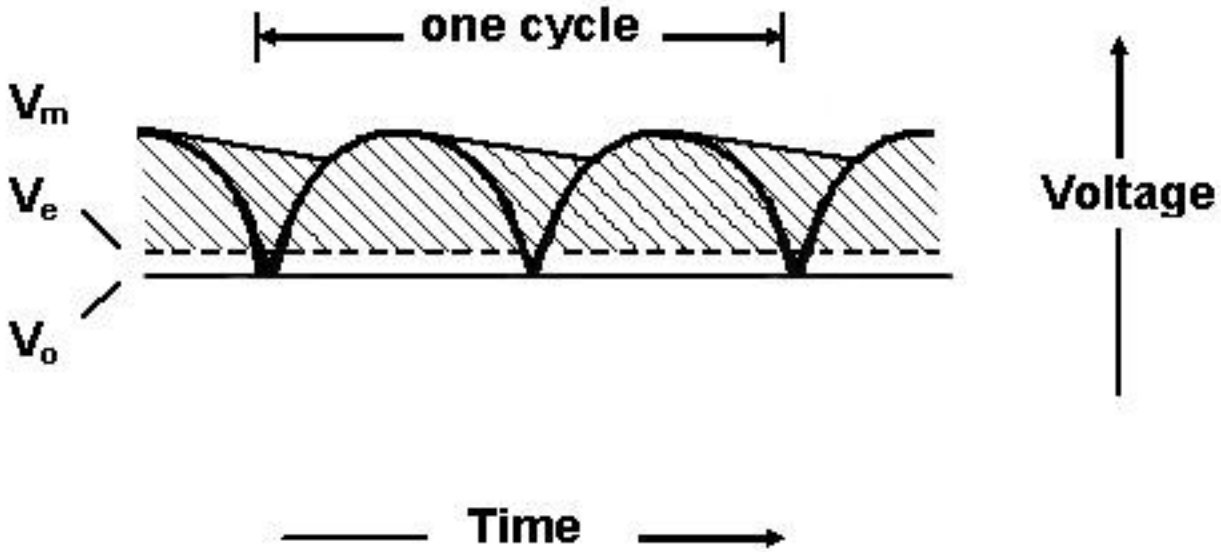


Instrumentation

A. X-ray Source Components

2. High-voltage generator

- Constant-potential



Instrumentation

A. X-ray Source Components

2. High-voltage generator

- Constant-potential

A smoothing is applied to the maximum value (V_m) for the whole cycle. The duty cycle approaches 90%.

There are still some ripple effects with constant-potential high voltage generators.

Instrumentation

A. X-ray Source Components

2. High-voltage generator

- High-frequency types

Converts a low frequency, low voltage input into a high frequency, low voltage waveform that produces a high frequency, high voltage output waveform.

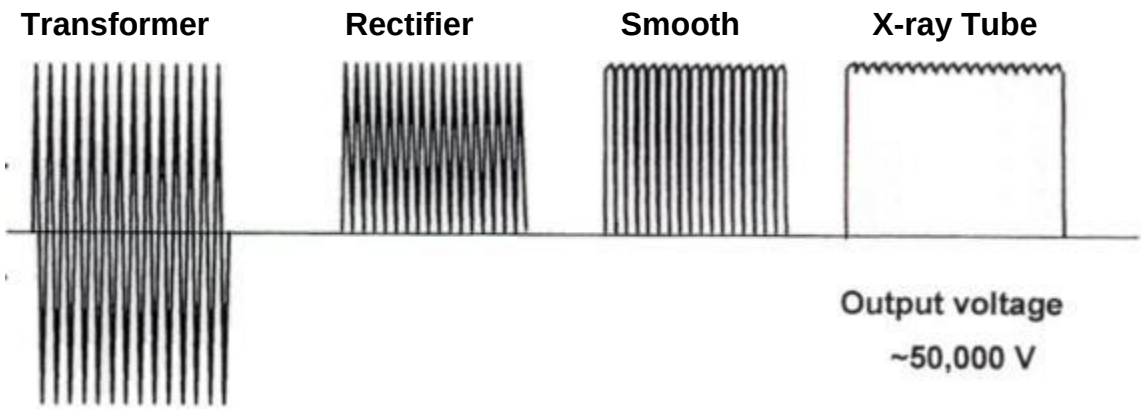
Instrumentation

A. X-ray Source Components

2. High-voltage generator

- High-frequency types

AC input power converted by rectification and smoothing to a low voltage DC waveform. An inverter circuit "chops" the DC into a high frequency AC square wave. AC square wave is input into a high-voltage transformer to produce a high voltage, high frequency AC waveform.

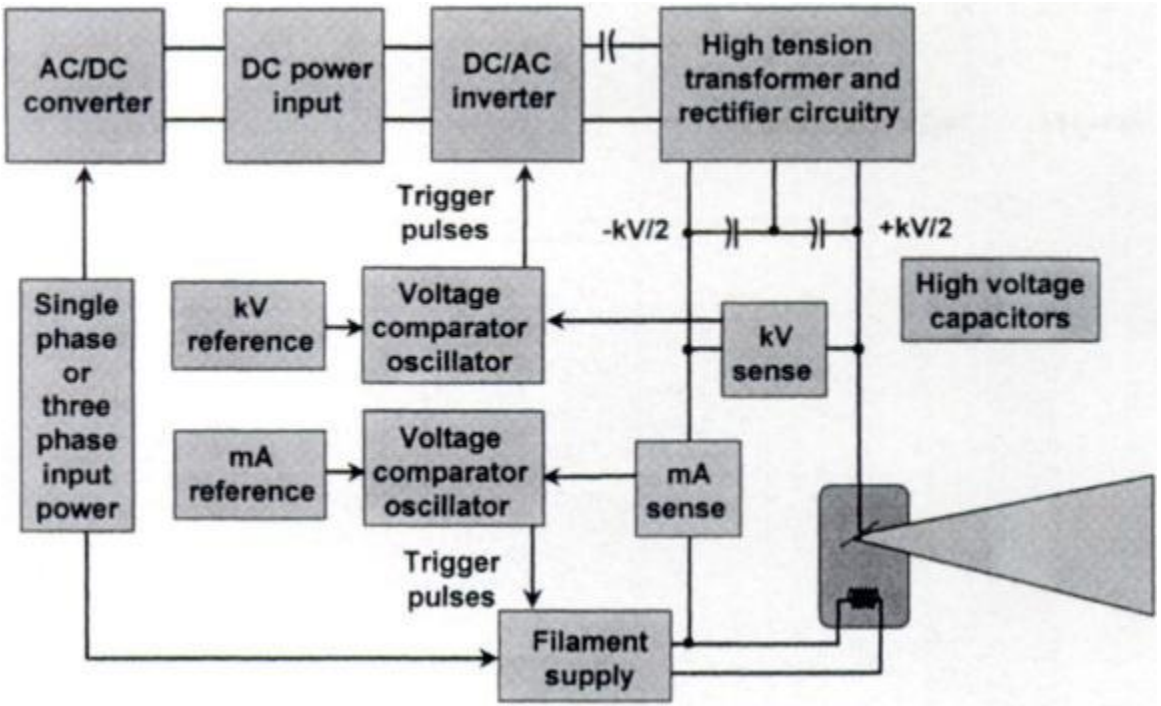


Instrumentation

A. X-ray Source Components

2. High-voltage generator

- High-frequency types



Instrumentation

A. X-ray Source Components

2. High-voltage generator

- High-frequency types

Advantages:

- can use either single- or three-phase input lines

- more efficient, more compact, less costly

- easy to repair

Instrumentation

A. X-ray Source Components

2. High-voltage generator

Generator Type	kV ripple
-Single-phase 1-pulse (self rectified)	100%
-Single-phase 2-pulse (full wave rectified)	100%
-3-phase 6-pulse	13-25%
-3-phase 12-pulse	3-10%
-Medium high frequency inverter	4-15%
-Constant potential	<2%

Instrumentation

A. X-ray Source Components

2. High-voltage generator

The transformers supplies filament current (i) and high voltage to the x-ray tube. All the components in the generator require high electrical insulation and are usually mounted in a high-dielectric oil-filled tank.

Instrumentation

A. X-ray Source Components

2. High-voltage generator

Output from an x-ray tube powered by high-voltage generator is described by radiation flux.

Flux – density of x-ray photon per unit area per second.

Takeoff angle – angle between the plane of the tube target and an incident slit of an experiment.

Instrumentation

A. X-ray Source Components

2. High-voltage generator

Goal is to use the maximum available flux from the x-ray tube, this is determined by:

1. **Maximum power rating (mA x kV) of the tube.**
2. **Type of generator employed.**
3. **Optimum kilovolt level.**
4. **Takeoff angle of x-ray tube.**
5. **Choice of monochromatic conditions.**
6. **Desired lifetime of the tube.**

Instrumentation

A. X-ray Source Components

2. High-voltage generator

The optimum choice of V and i can be determined from an isowatt curve, plot of operating voltage vs total x-ray intensity from the tube.

Instrumentation

A. X-ray Source Components

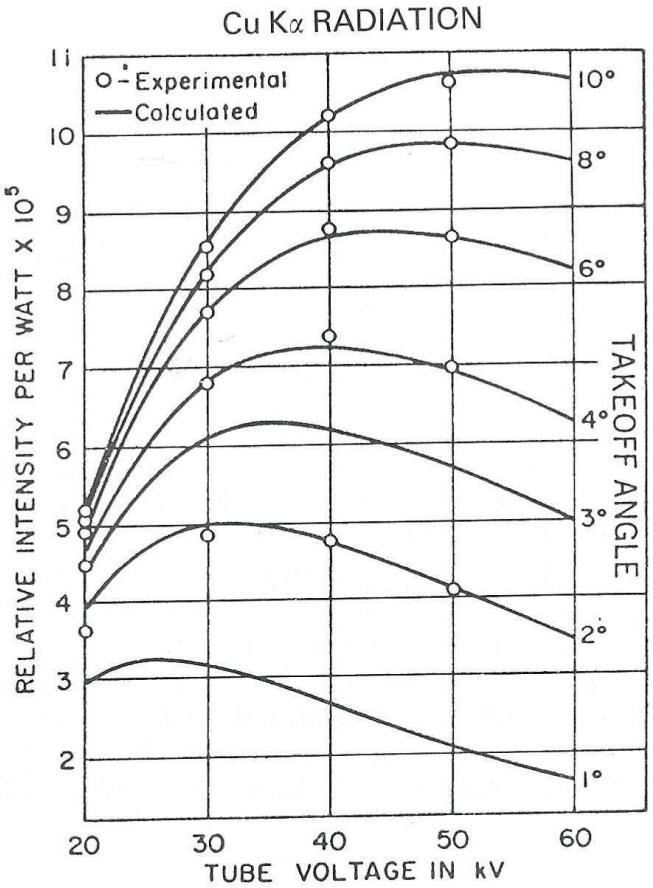


Figure 4.5. Experimental and calculated isowatt curves for a copper target X-ray tube. [illegible] from Short [5, p. 569], with permission.

Instrumentation

A. X-ray Source Components

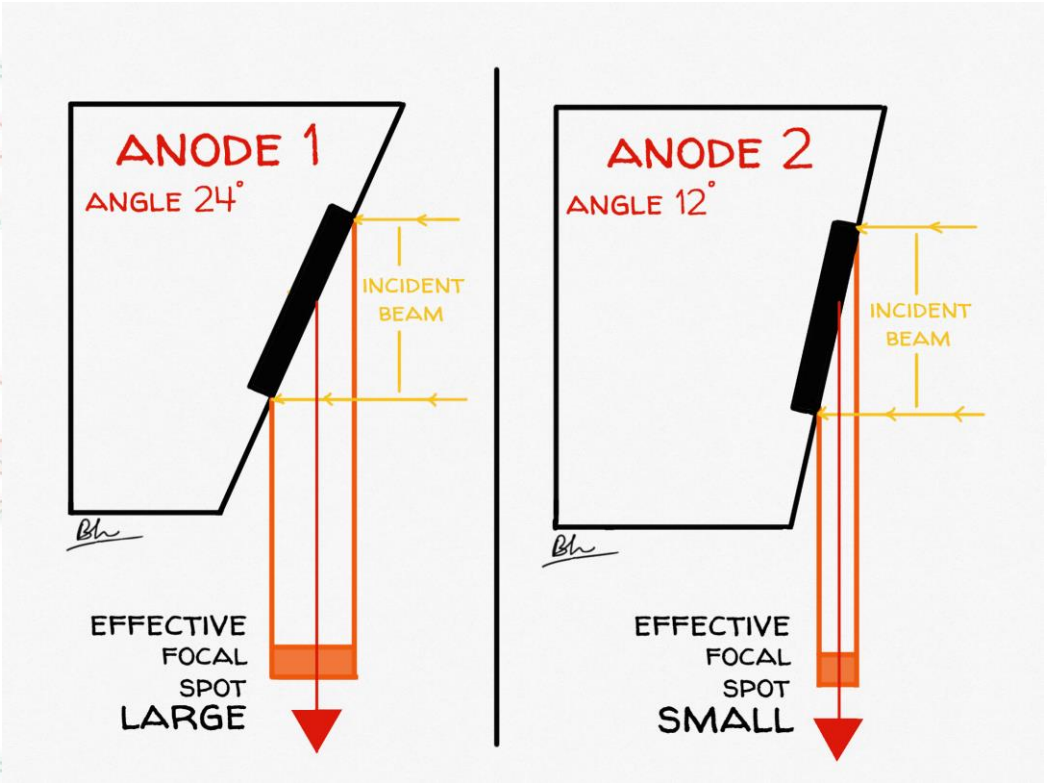
X-ray take off angle

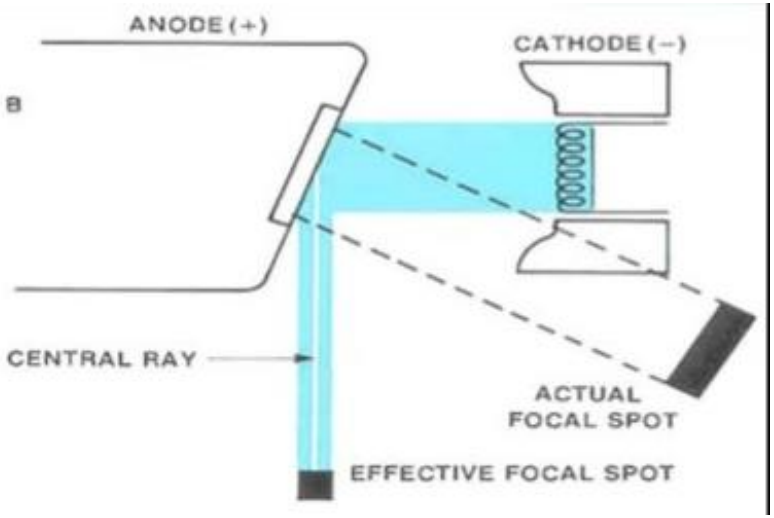
- **Take off angle is generally set to six degrees.**
- **There are two types of focal spots, a line focus and a point focus, depending on the take off direction.**

Instrumentation

A. X-ray Source Components

X-ray take off angle





Instrumentation

A. X-ray Source Components

2. High-voltage generator

Also must remain within the power curve of a given tube.

Instrumentation

A. X-ray Source Components

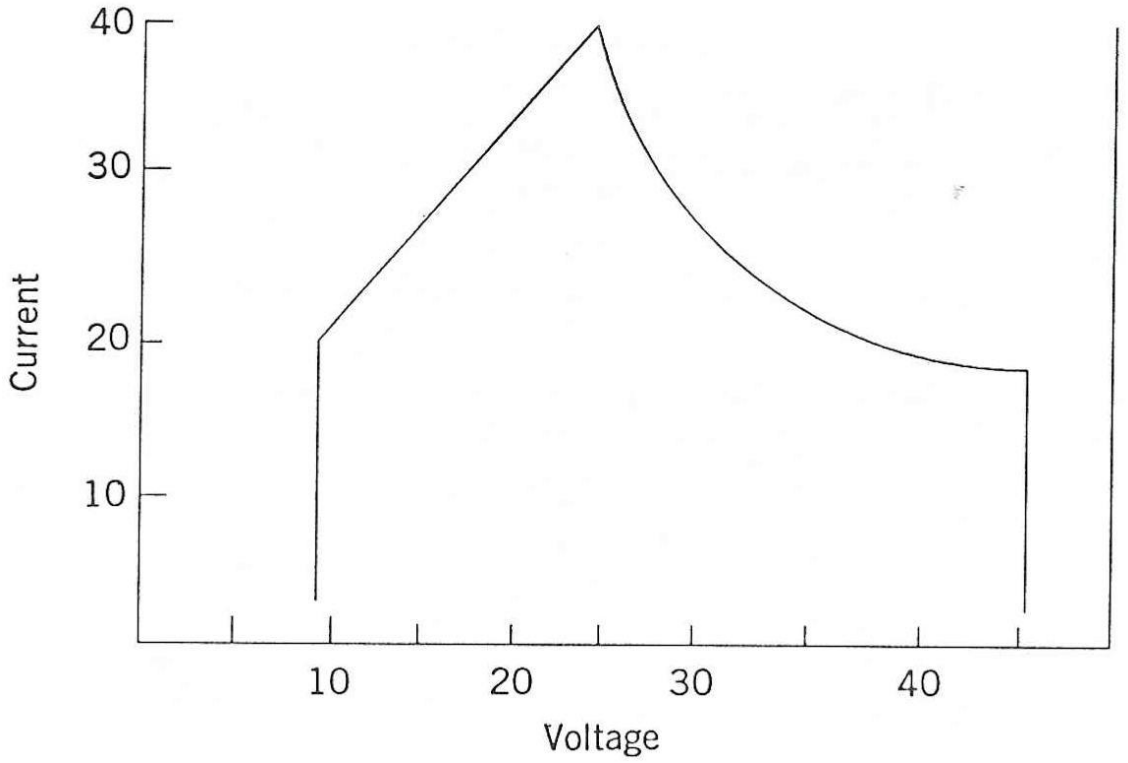


Figure 4.6. Power curve for an X-ray tube.

Instrumentation

A. X-ray Source Components

3. Source Stability

Drift - variation in output of the source.

There are several types:

Type	Time	Magnitude(%)	Source
Ultralong	mons/yrs	1 - 20	Aging of the tube
Long	days/wks	0.2 - 0.5	Thermal, focal spot wander
Short	mins/hrs	less than 0.1	Stabilization circuit
Ultrashort	msec	0.2 - 10	Transients

Instrumentation

A. X-ray Source Components

4. Specific Loading

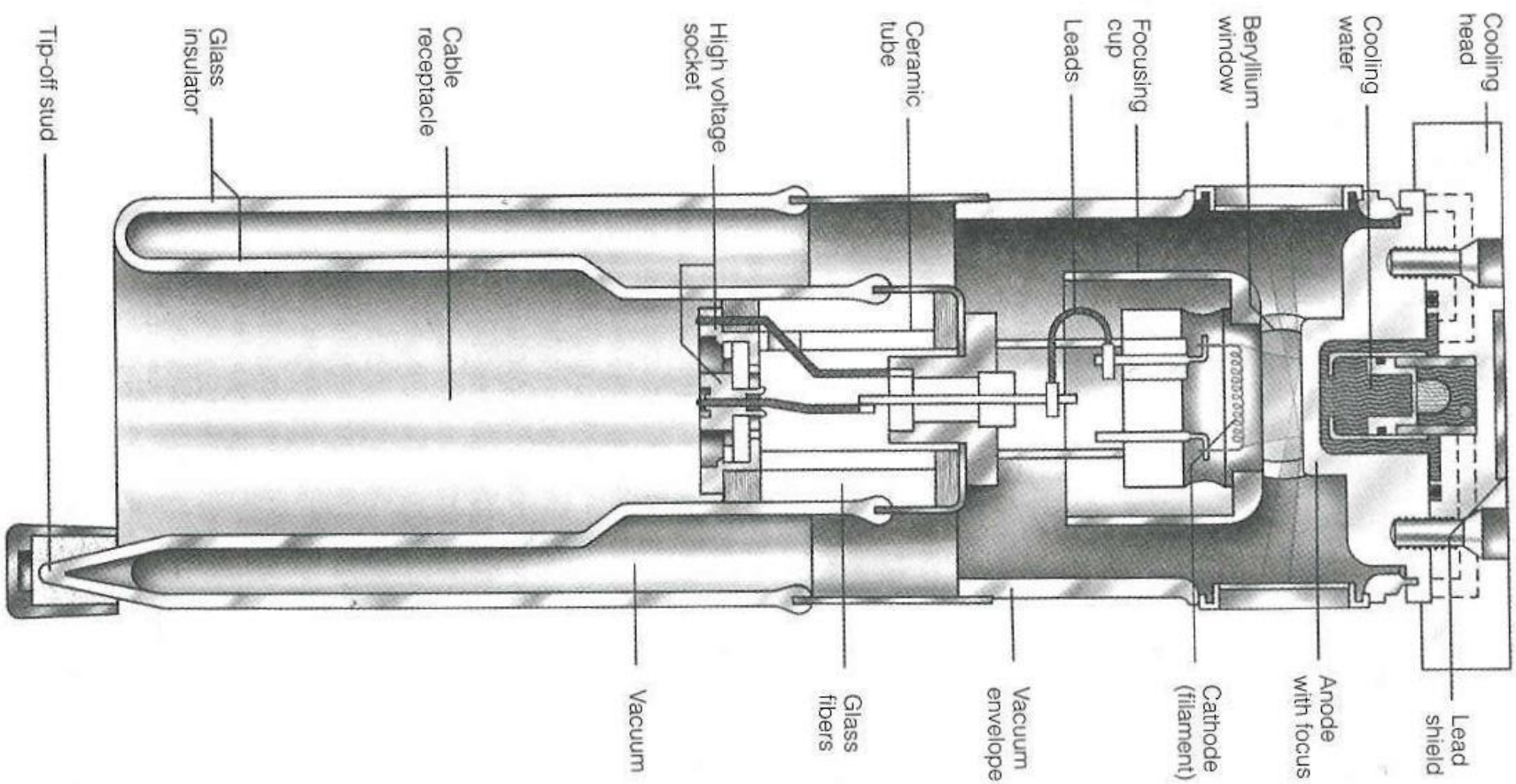
The focal spot for the normal x-ray beam is ~ 1 x 10 mm.

Microfocus tubes spot size is about 0.1 x 1 mm - used for high resolution work.

Maximum rating of the x-ray tube depends upon the ability of the anode to dissipate heat.

Instrumentation

A. X-ray Source Components



Instrumentation

A. X-ray Source Components

4. Specific Loading

The specific loading (W/mm^2) of the anode is rated for tubes.

Tube type	Dimensions (mm)	Loading (kW)	Specific Loading(W/mm^2)
Fine focus	0.5 x 12	2.0	333
Normal focus	1.0 x 12	2.5	208
Broad focus	2.0 x 12	3.0	125
Rotating Anode	0.5 x 10	15.0	3000

Instrumentation

A. X-ray Source Components

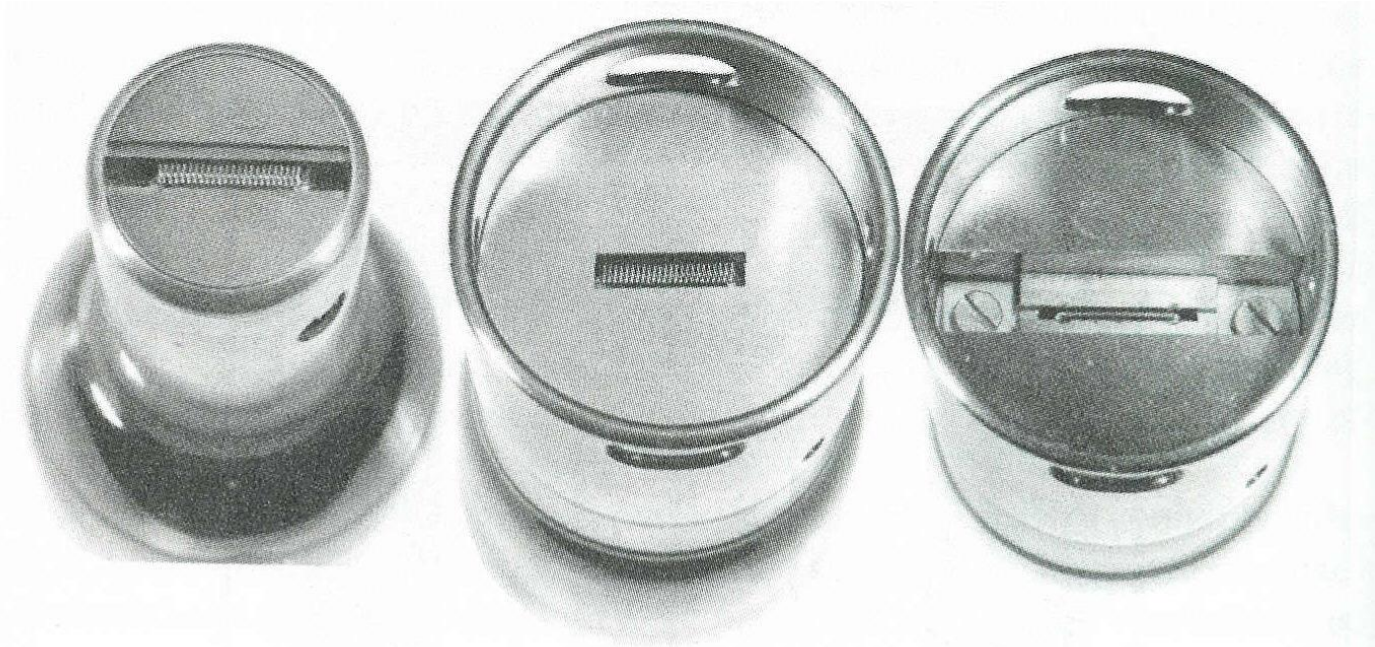


Figure 4.8. X-ray tube filament assemblies: (a) broad focus; (b) normal focus; (c) fine focus. Reprinted from R. Jenkins and J. L. de Vries, *An Introduction to Powder Diffractometry*, p. 19, Fig. 23. Copyright © 1977, N. V. Philips, Eindhoven, The Netherlands.

Instrumentation

A. X-ray Source Components

X-ray tube care

New and unused x-ray tubes require a running-in period before use at full loading.

When made, air must all be removed to prevent oxidation of the tube filament.

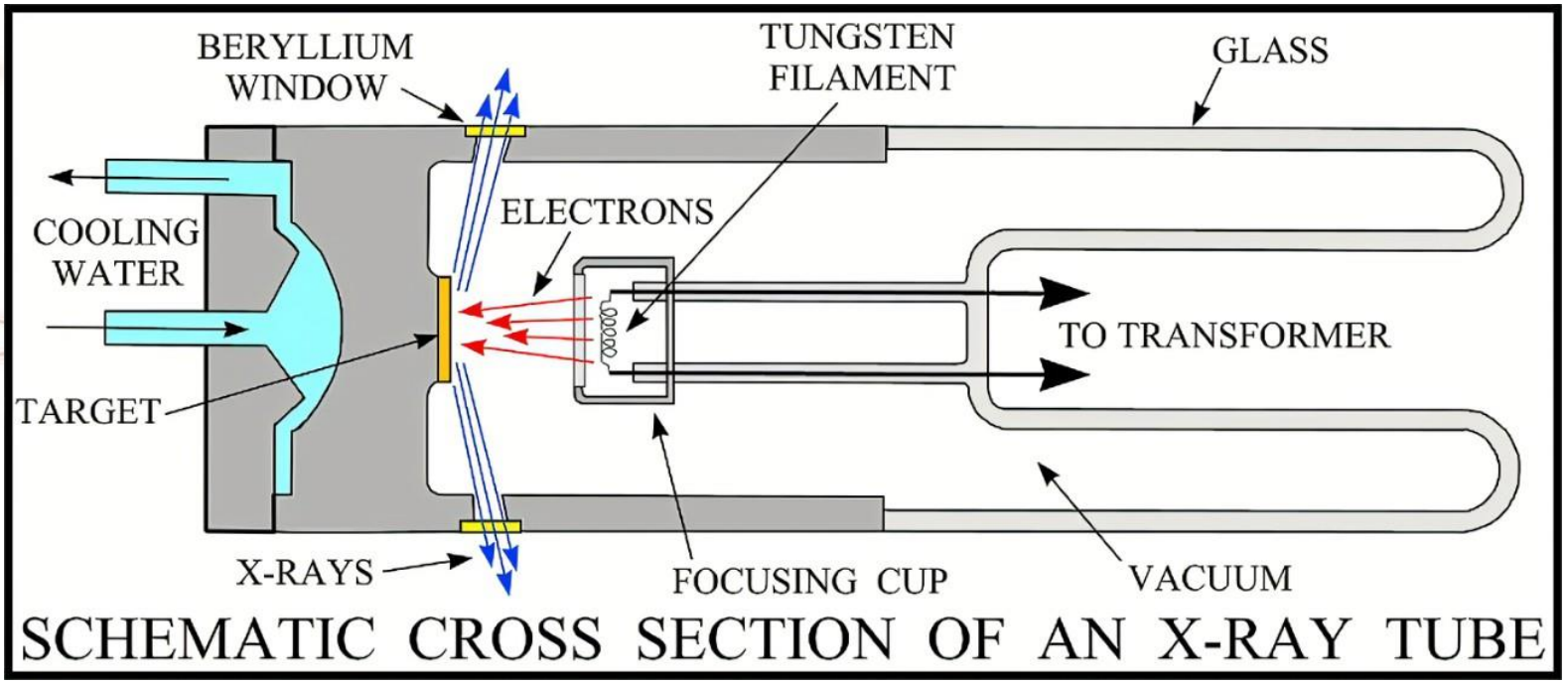
The space charge must be maintained to keep air on walls by a static charge.

Most common cause of tube breakdown is failure of cooling system. (Also must keep shower head clean)

Instrumentation

A. X-ray Source Components

Chiller is critical for keeping tube cool.



SCHEMATIC CROSS SECTION OF AN X-RAY TUBE

Instrumentation

A. X-ray Source Components

The x-ray source should be spectrally pure, however spectral contamination can cause the addition of weak unwanted lines in the pattern. Contamination sources include:

Element	Specific source	Effect
Cu	Anode block	increase w/ time
W	Filament	increase w/ time
Fe	Window seal	generally small
Ca	Window	generally small

Instrumentation

A. X-ray Source Components

5. Rotating anode

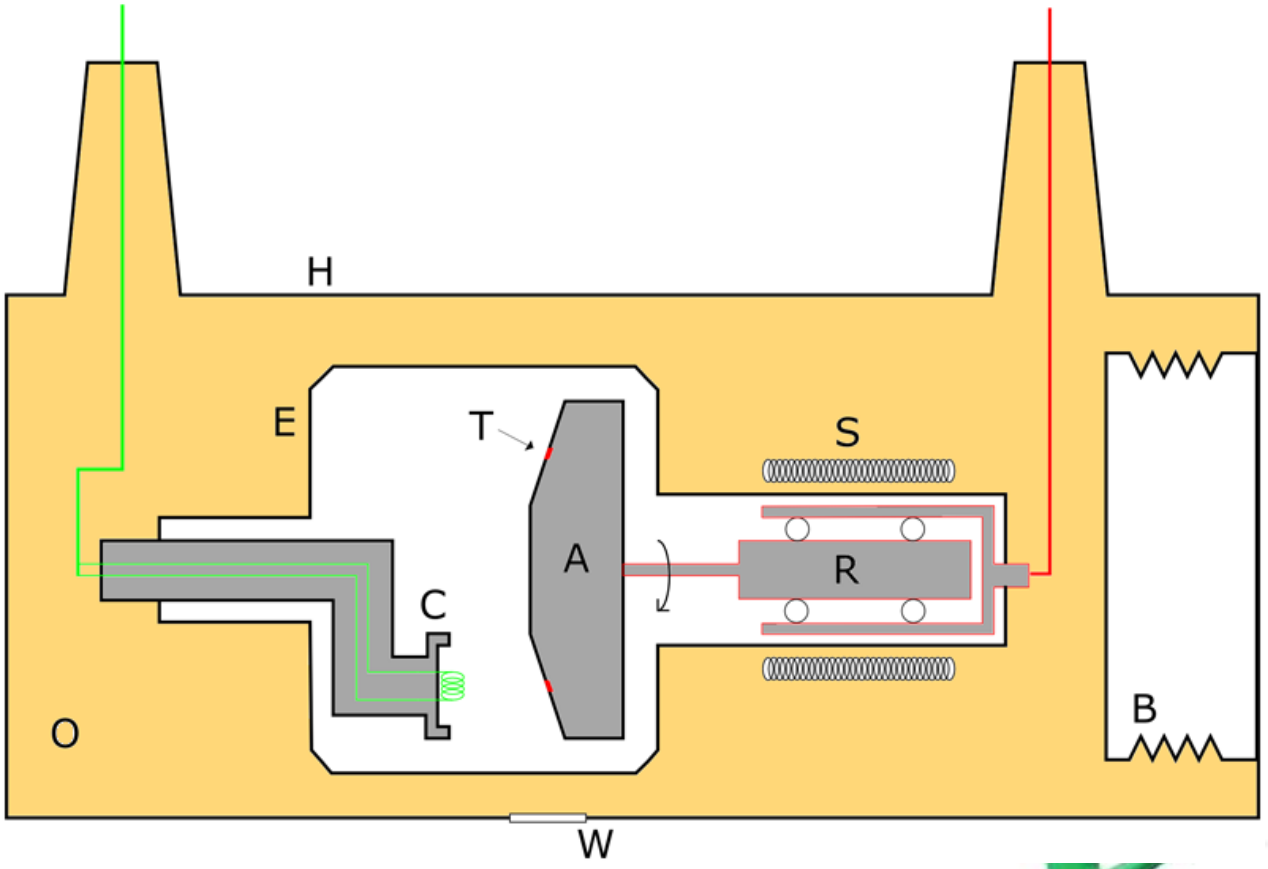
Anode rotated at high speed - allows higher amperage with better cooling - increases intensity of the x-ray tube.

Problems include mechanical difficulties of having a high-speed motor drive that must feed through a vacuum. Must use ferro-fluidic seals and turbomolecular pumps.

Instrumentation

A. X-ray Source Components

5. Rotating anode



Instrumentation

A. X-ray Source Components

5. Rotating anode

Instrumentation

Assignments:

Read Chapter 3, 4, 5, 9 and 13 from:

-Introduction to X-ray powder Diffractometry by Jenkins and Synder

Read Chapter 3, 4, 6, 13, and 14 from

-Elements of X-ray Diffraction by Cullity and Stock

Read Chapter 2 from Norton

Homework 4 Due 10/10/24 – Today

Homework 5 Due 10/17/24 – Solving a Cubic System

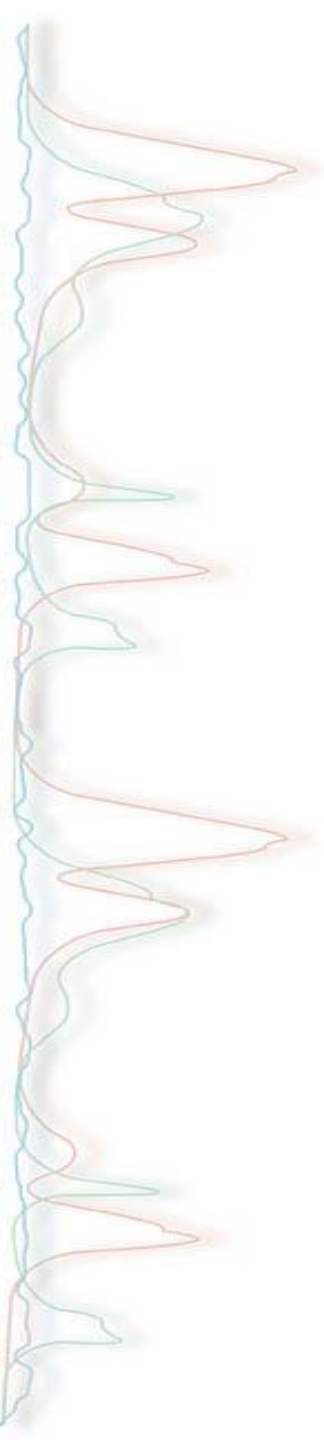
Group Assignments:

Group 1: Cr and Si
D.Perez, H.Castro, L.Ngo, A.M.Rodriguez MK.Altafi, T.Tran

Group 2: Cu and TiN
S.Nair, S.Amagbor, S.Sarang, S.Guraja, D.Kumaravel

Group 3: Fe and TiN
R.McCrary, T.Agbara, S.Hemmati, N.Stewart, D.Ballew

Group 4: Ni and Si
R.Demmelash, J.Castillo, A.Outha-Aphay, S.Hanlon O.Ayeni



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