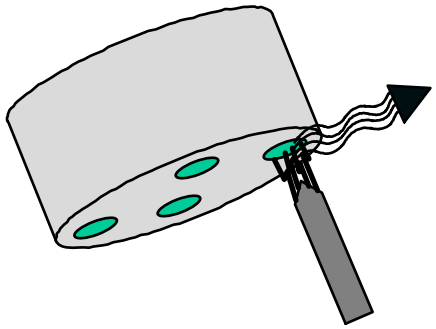
Chemistry and Chemical Sampling

Updated 2012



Chemical Analysis

"The Quality of an Analysis is Never Better than the Quality of the Sample"

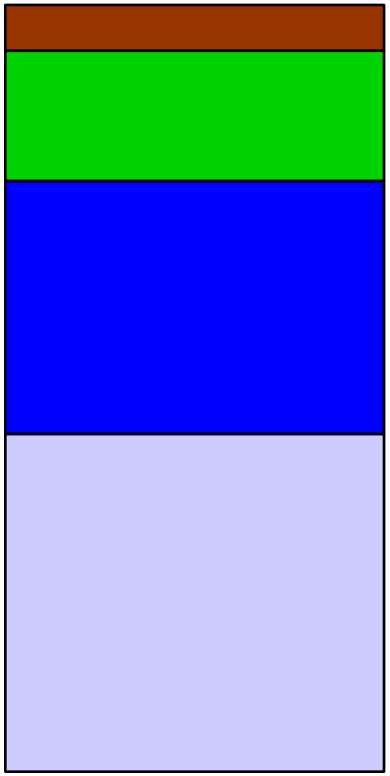
Chemistry and Chemical Sampling

Why is metal chemistry control important?

- Chemistry directly affects mechanical properties
- Mechanical properties directly affect the performance of our sheet products
- On target with minimum variation chemistry reduces product variation

OES Analytical Error

Relative contributions



6% Method

17% Instrument Drift

33% Calibration Standard

44% Sample **(sample preparation and sampling)**

Sample Defect Identification

- Defective samples adversely affect chemical analysis
- Defective samples adversely affect automated spectrometer operation

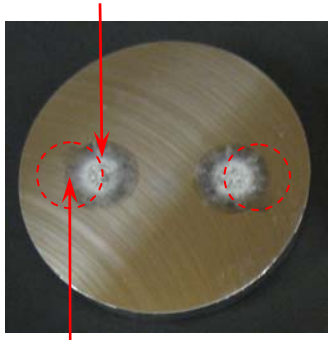
The Greatest Source of Error

Question: What is the greatest source of error in Quantometer (OES) Analysis?

Answer: The Sample (sampling procedure, sample quality and sample preparation)

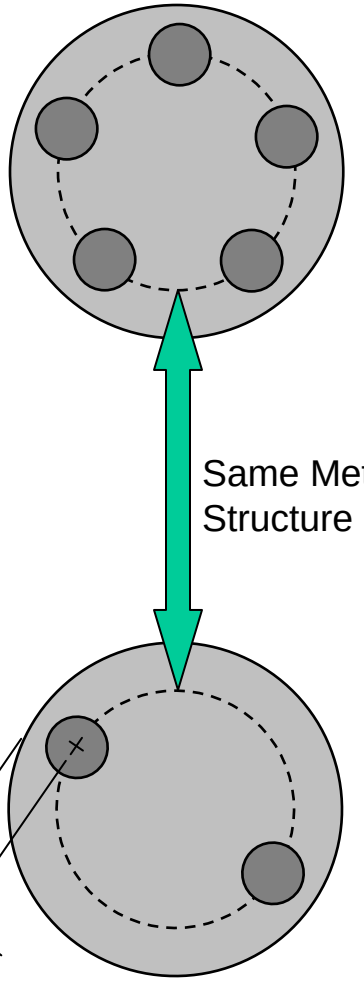
Best Practice: Spark Location and Quantity

Samples of 3xxx alloy must have at least 2 good sparks evenly spaced. Samples of 5182 must have at least 3 good sparks evenly spaced. Sparks on standards and production samples should be approximately 3/8” in from the edge. Sparking samples or standards anywhere else is poor practice.



Improper spark center, too far from sample edge

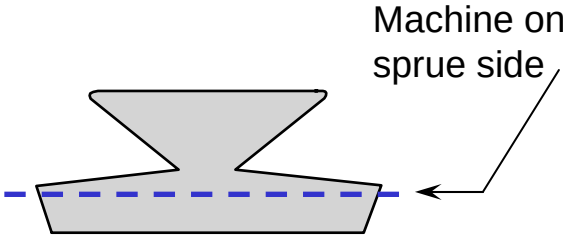
Proper spark center



Same Metallurgical Structure

8 -10 mm (3/8”)

OES Standard

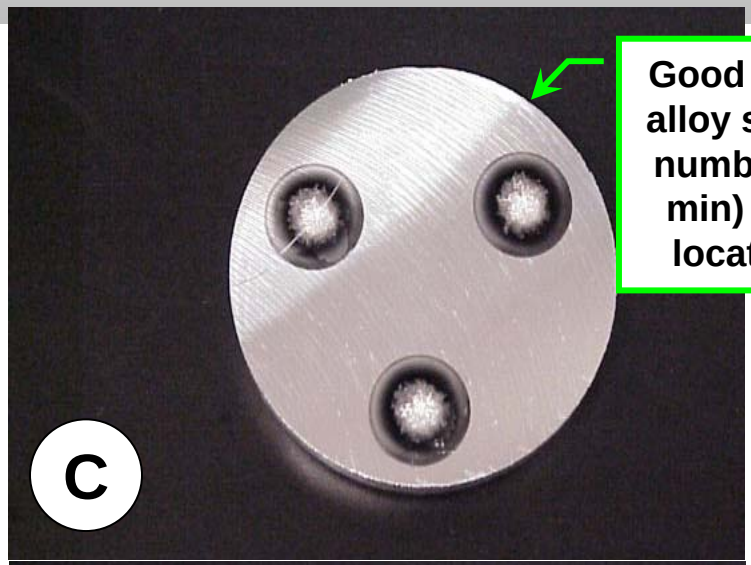


Machine on sprue side

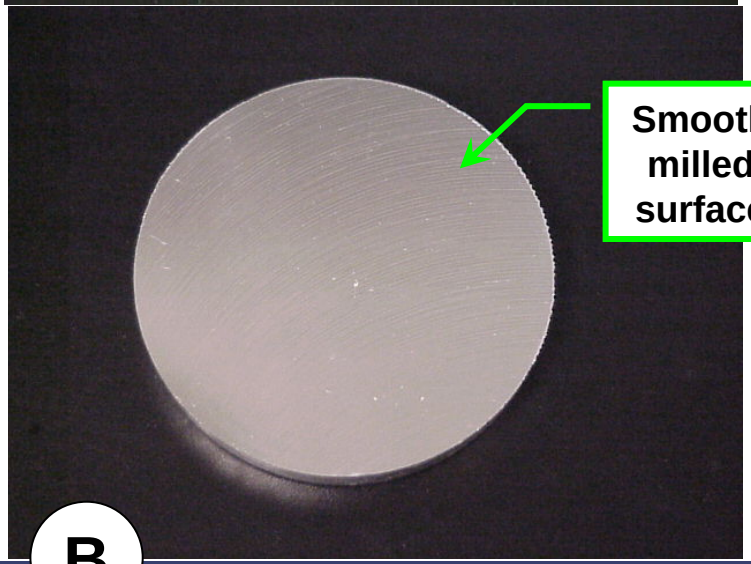
production sample
2 -1/4” diameter

Our Goal – A good sample

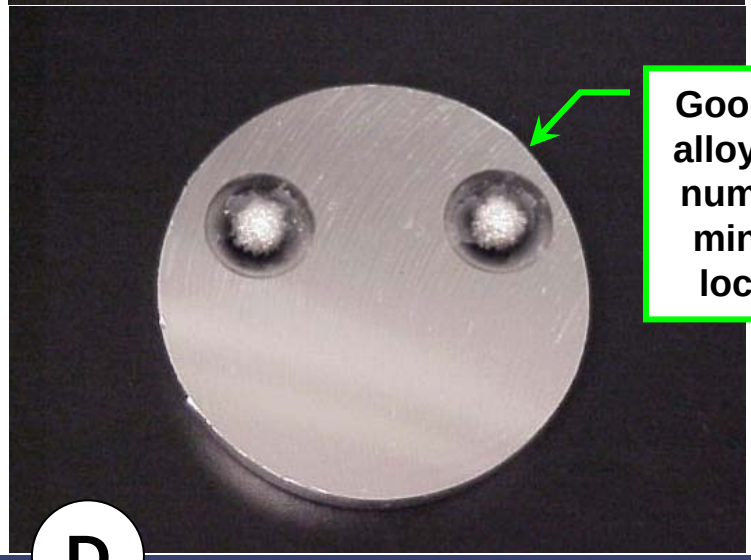
Good Sample – flat surfaces, square edges



Good 5xxx alloy spark number (3 min) and location

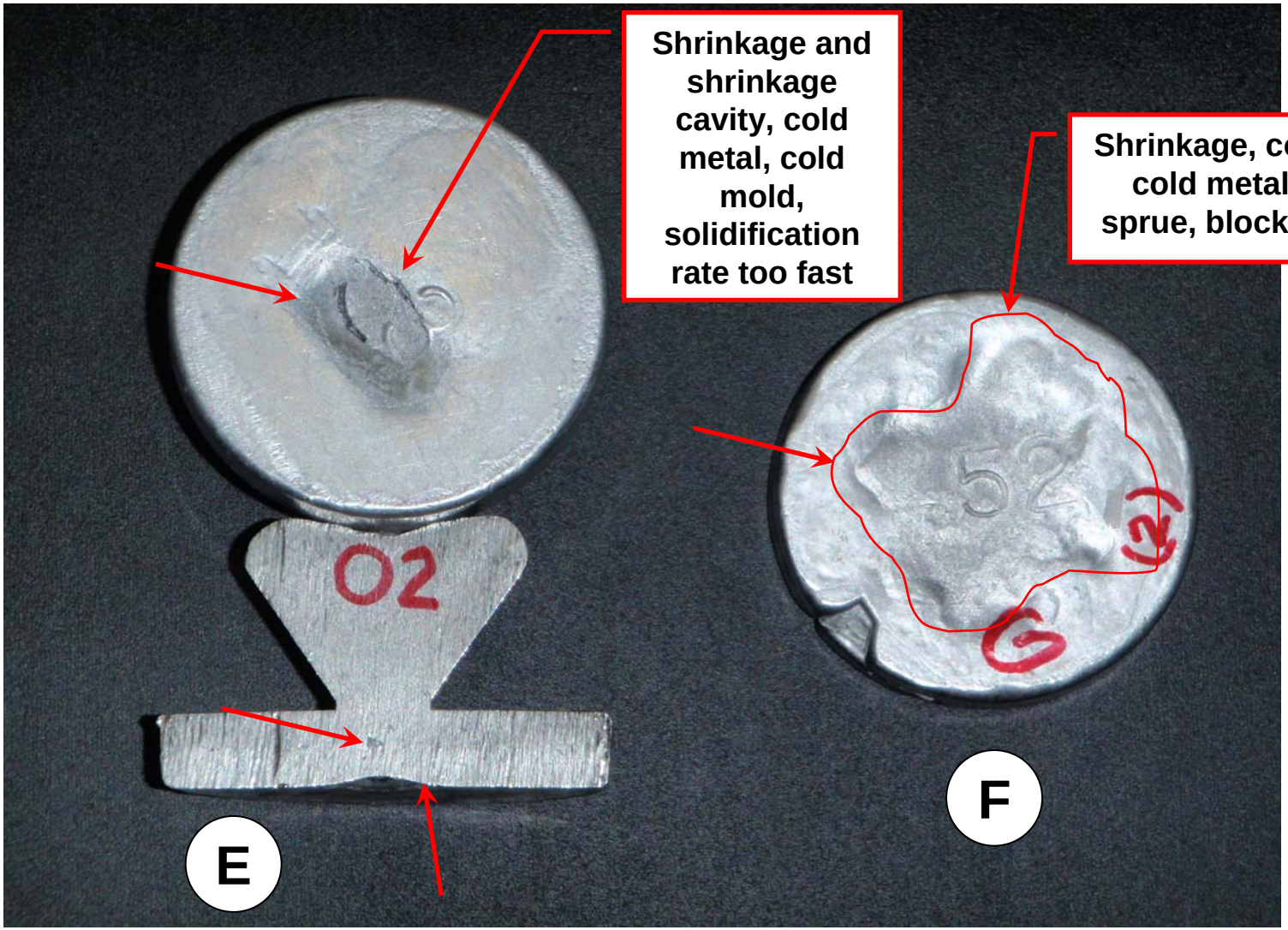


Smooth milled surface

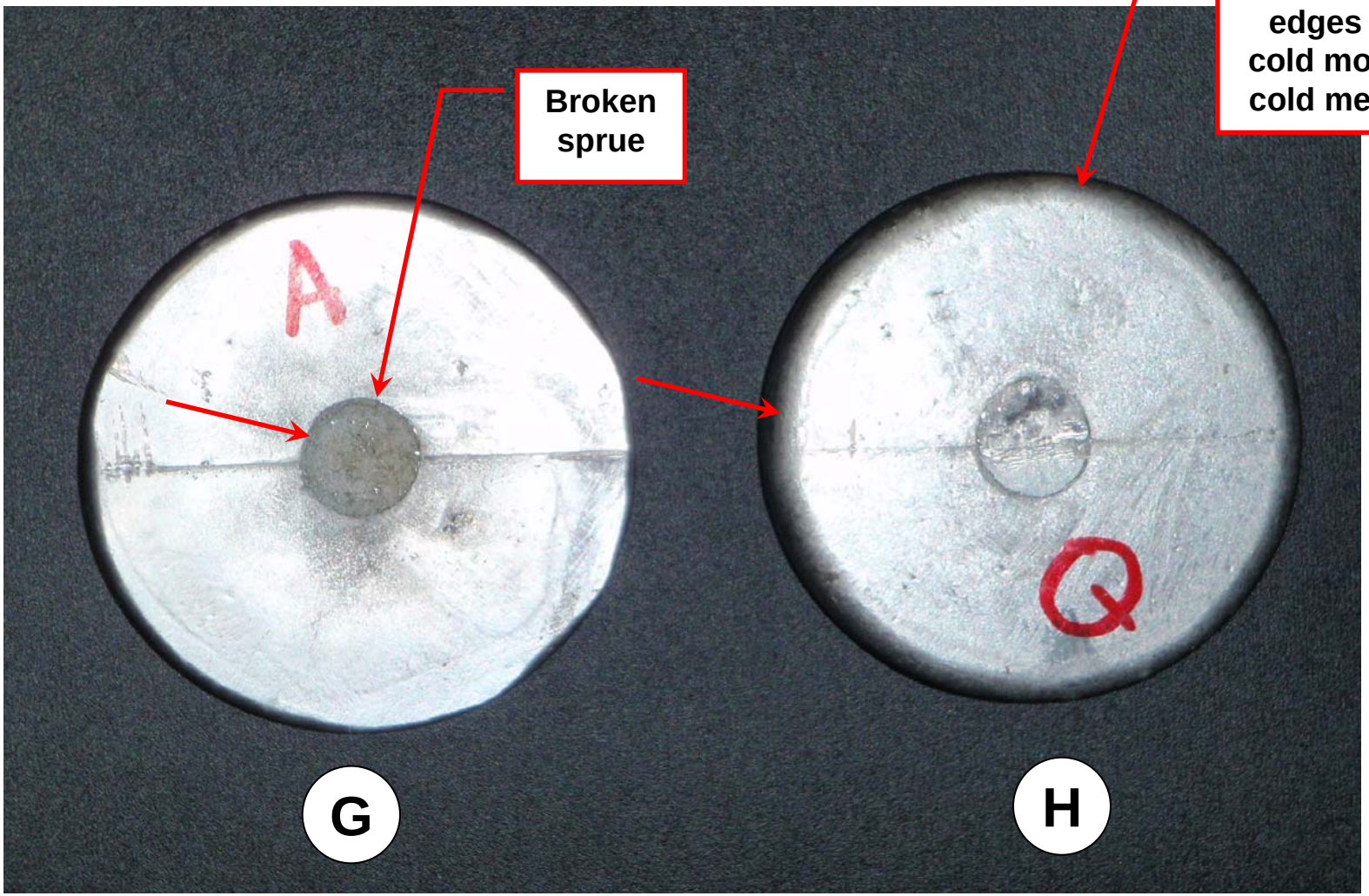


Good 3xxx alloy spark number (2 min) and location

Sample Defects

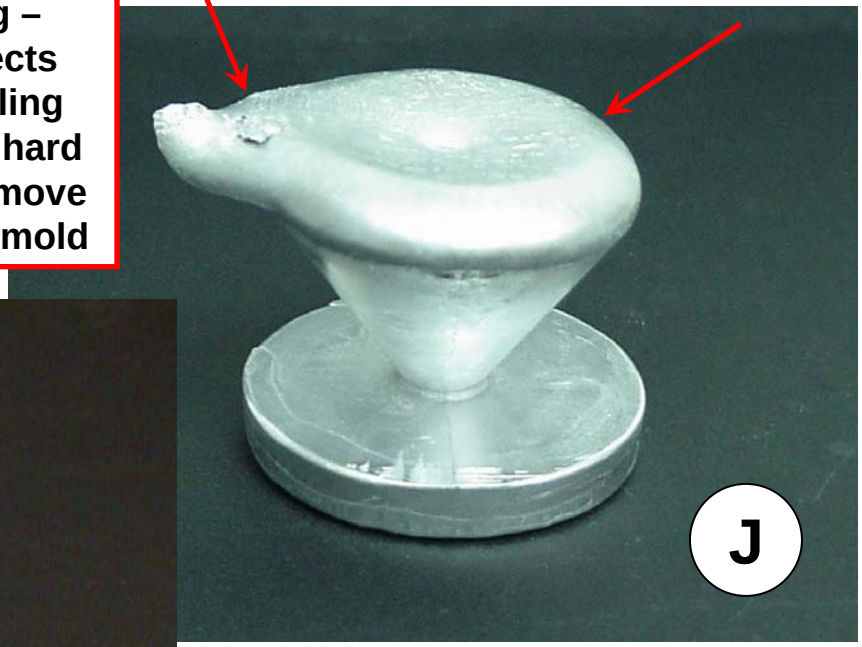


Sample Defects

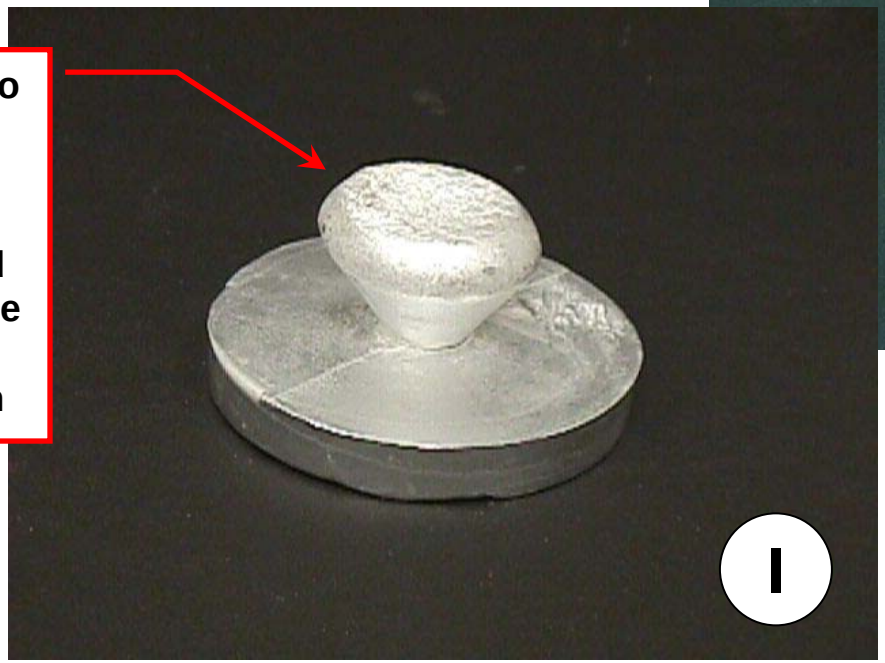


Sample Defects

Sprue too big – affects cooling rate, hard to remove from mold

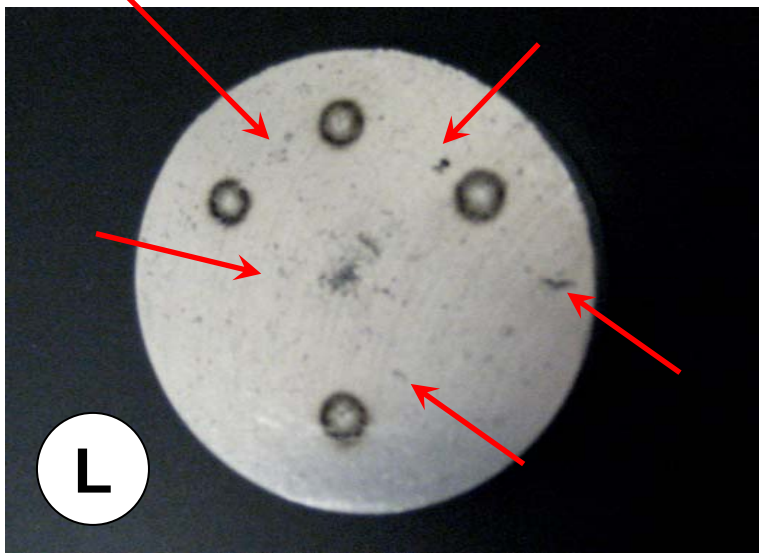


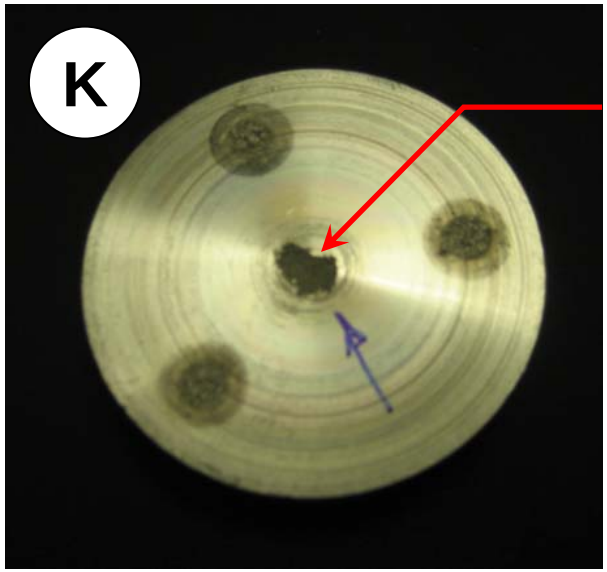
Sprue too small – affects cooling rate and shrinkage cavity location



Sample Defects

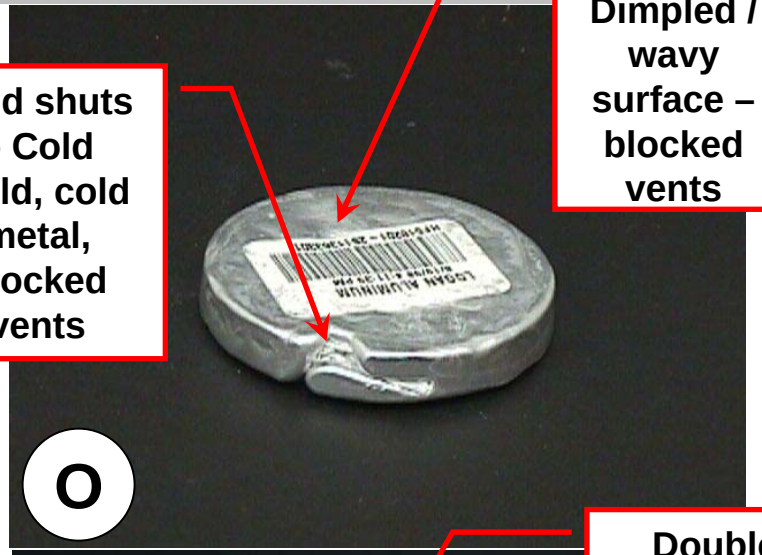
Dross leading to bad sparks

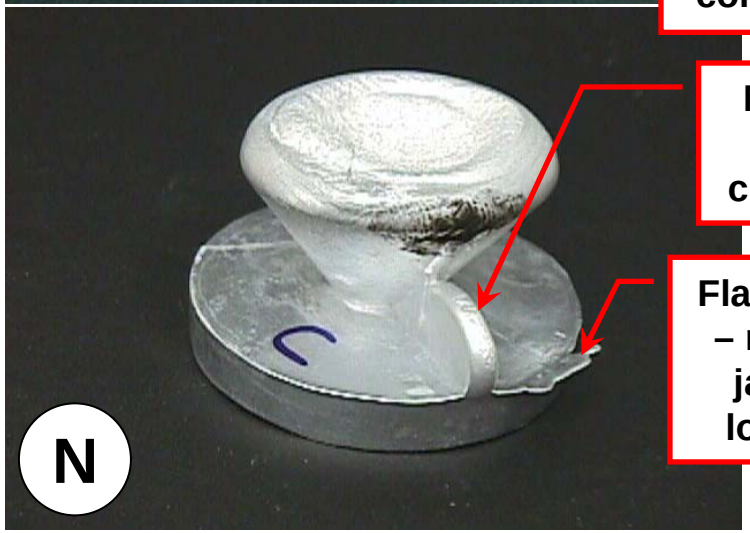




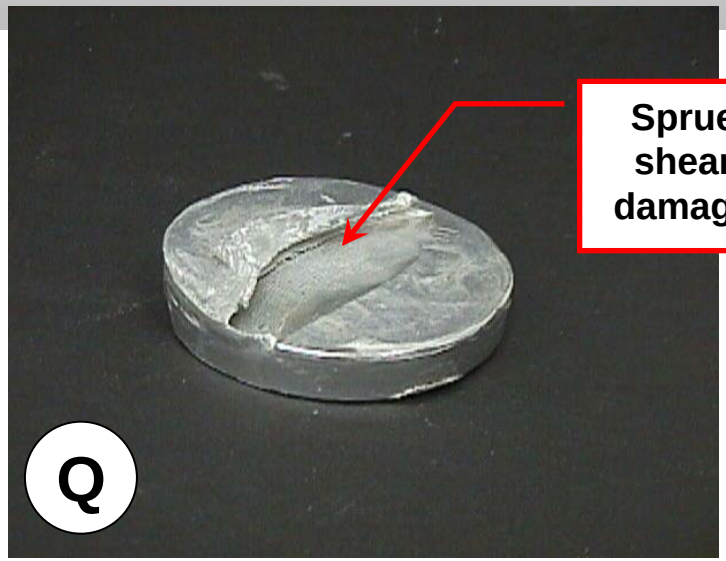
Shrinkage cavity from cold mold, cold metal, short sprue and/or sprue broken off before complete solidification

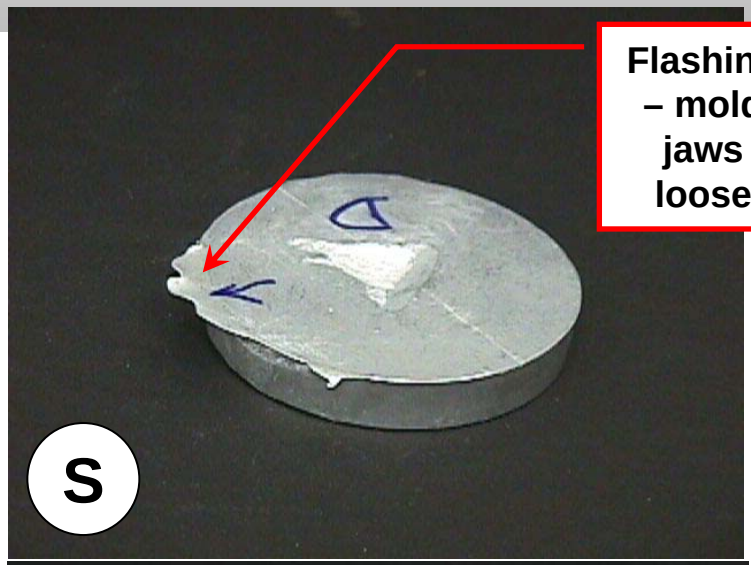
Sample Defects

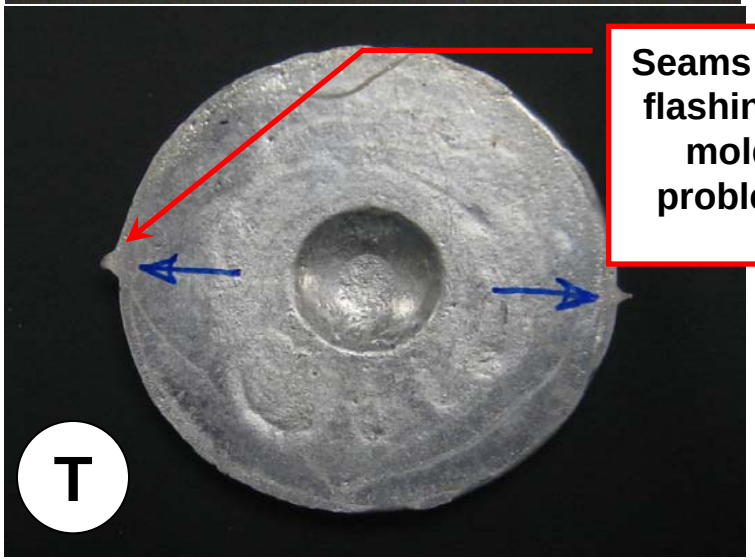




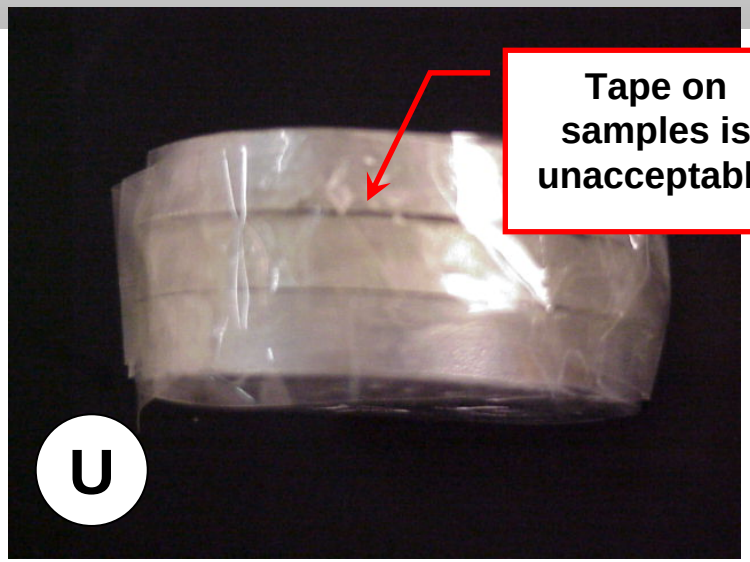
Sample Defects

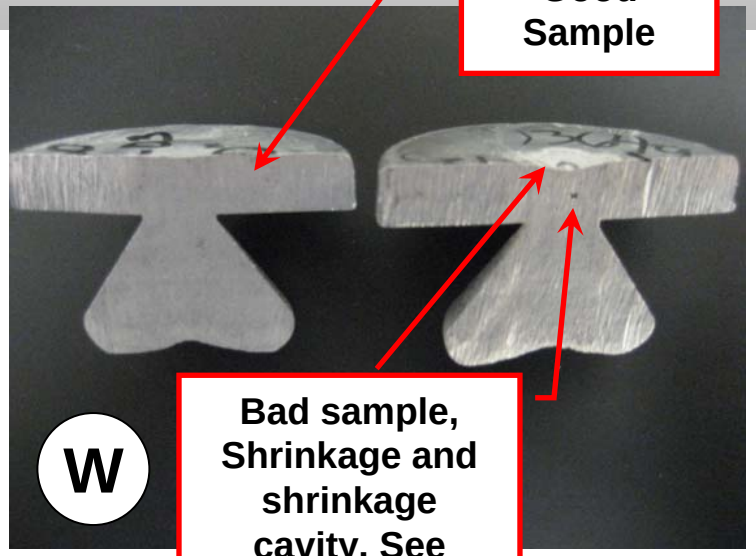


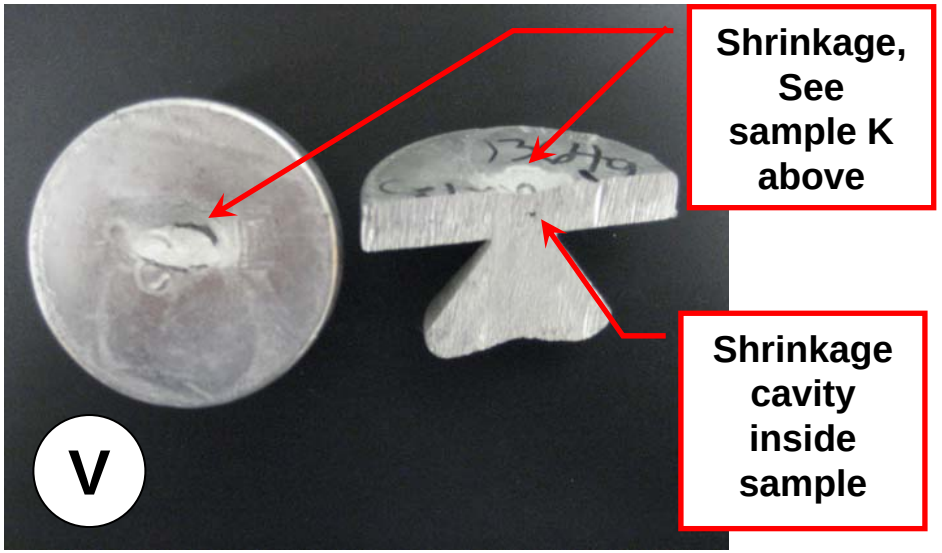


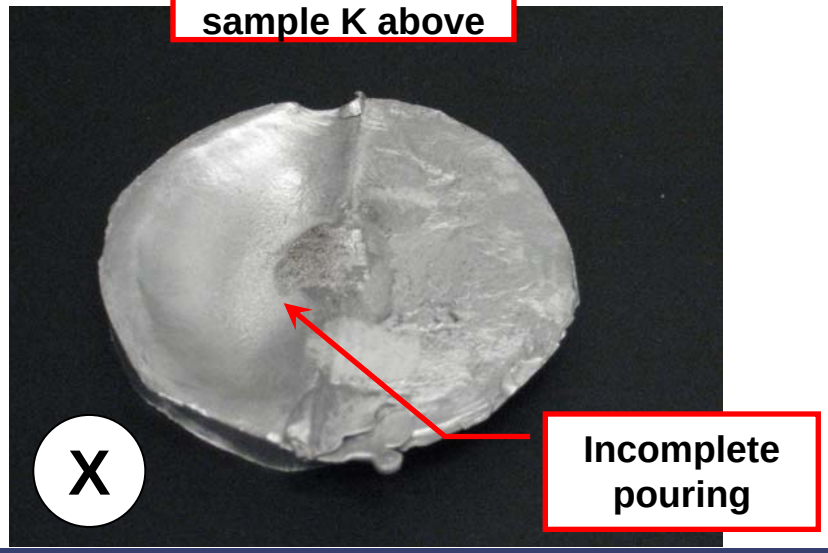


Sample Defects

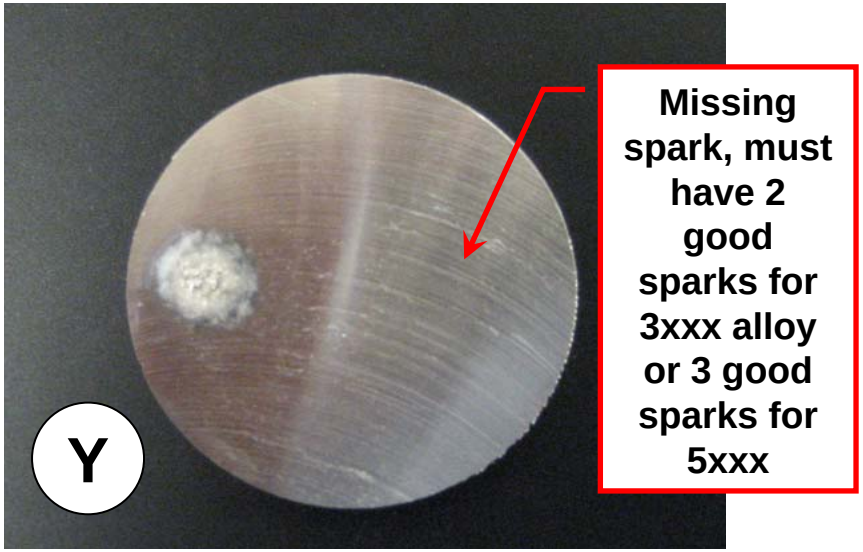


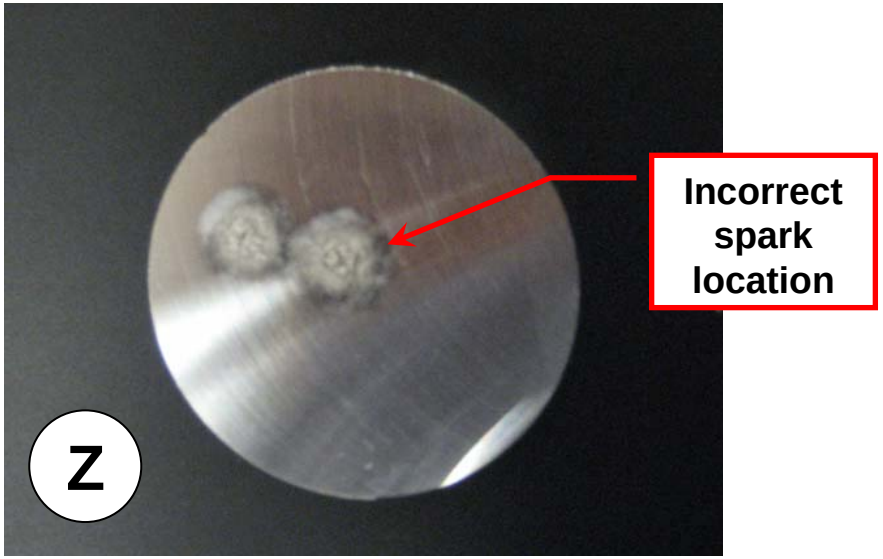






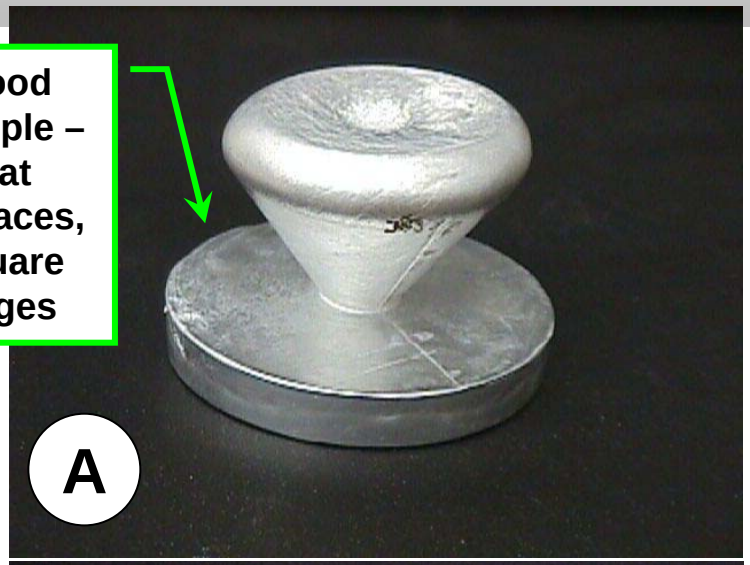
Sample Defects

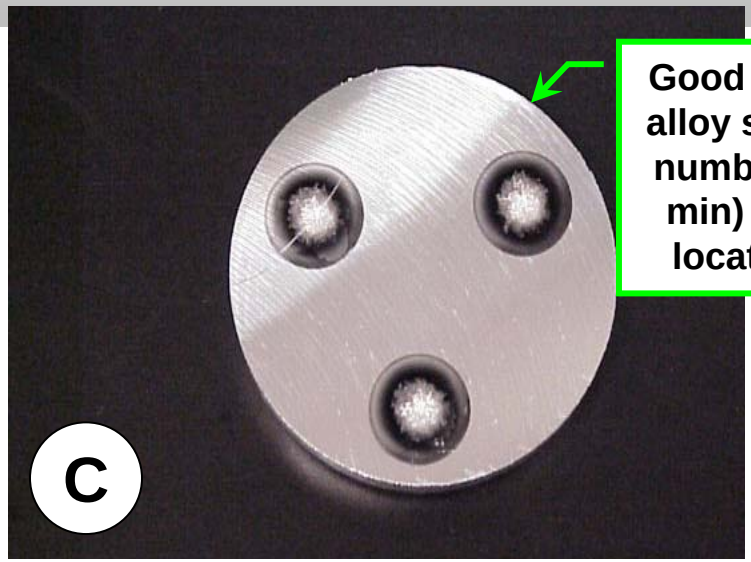




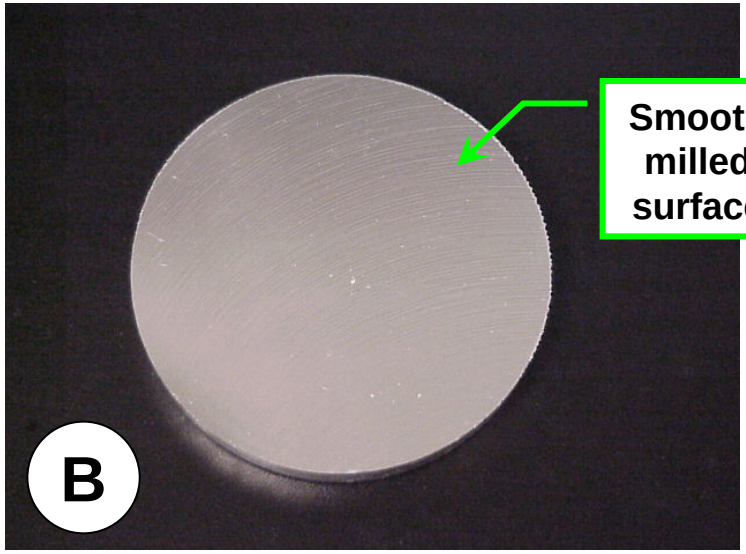
Our Goal – A good sample

Good Sample – flat surfaces, square edges

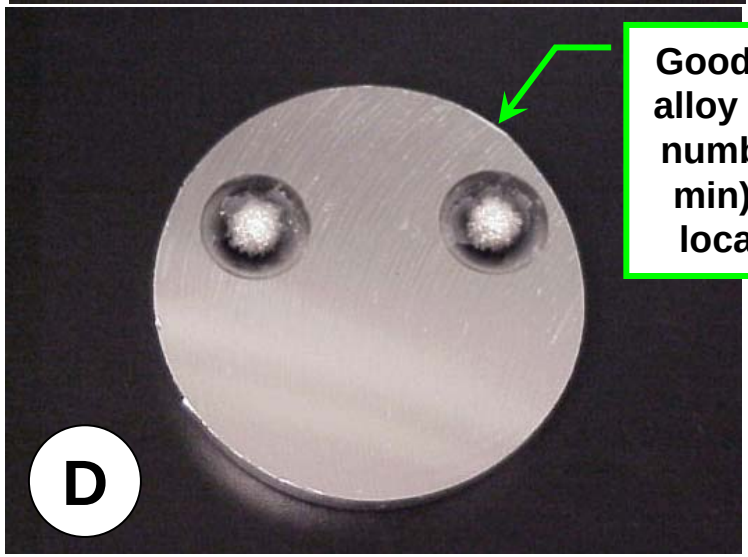




Good 5xxx alloy spark number (3 min) and location

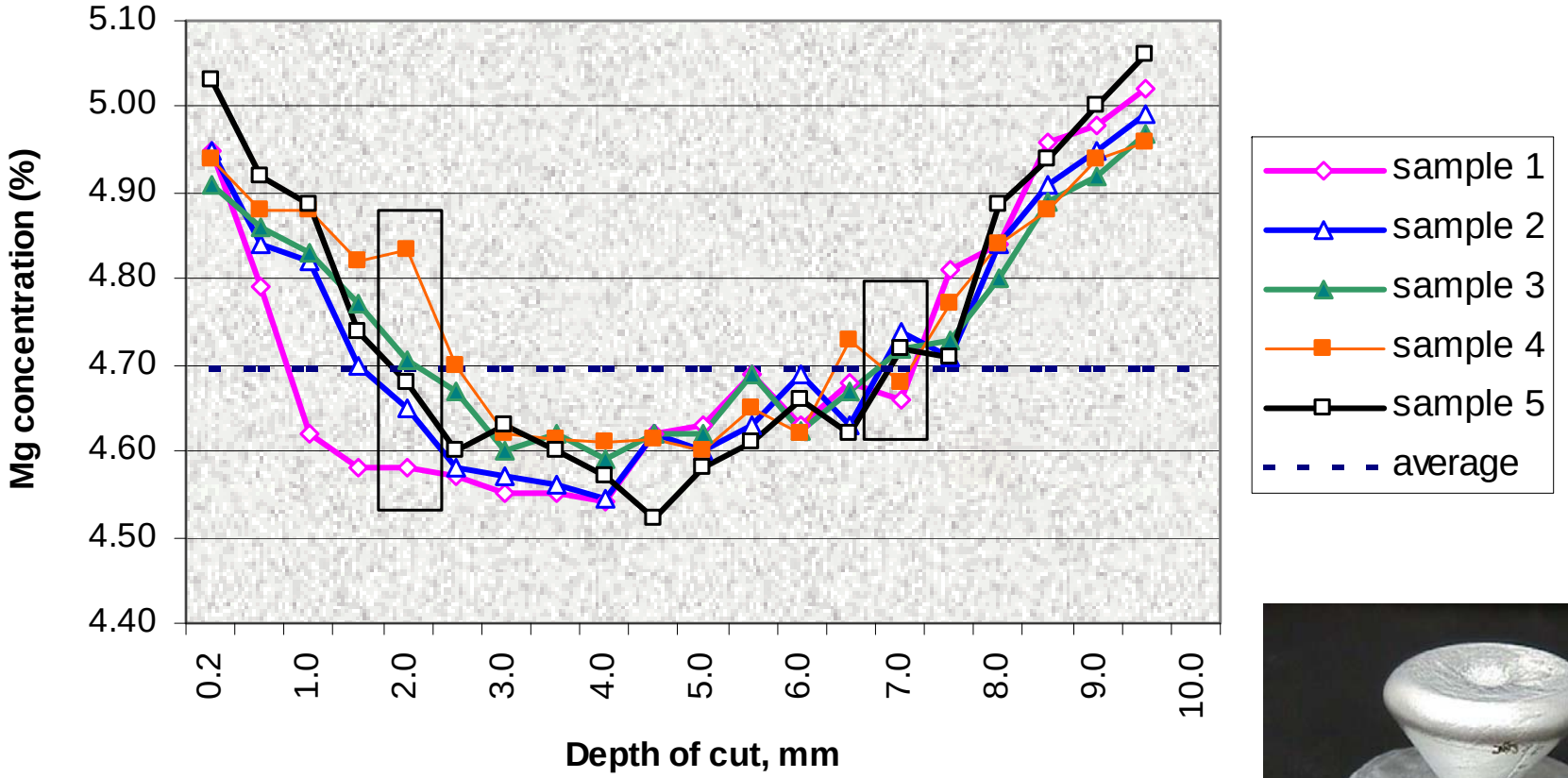


Smooth milled surface



Good 3xxx alloy spark number (2 min) and location

Mg Segregation vs. depth of cut

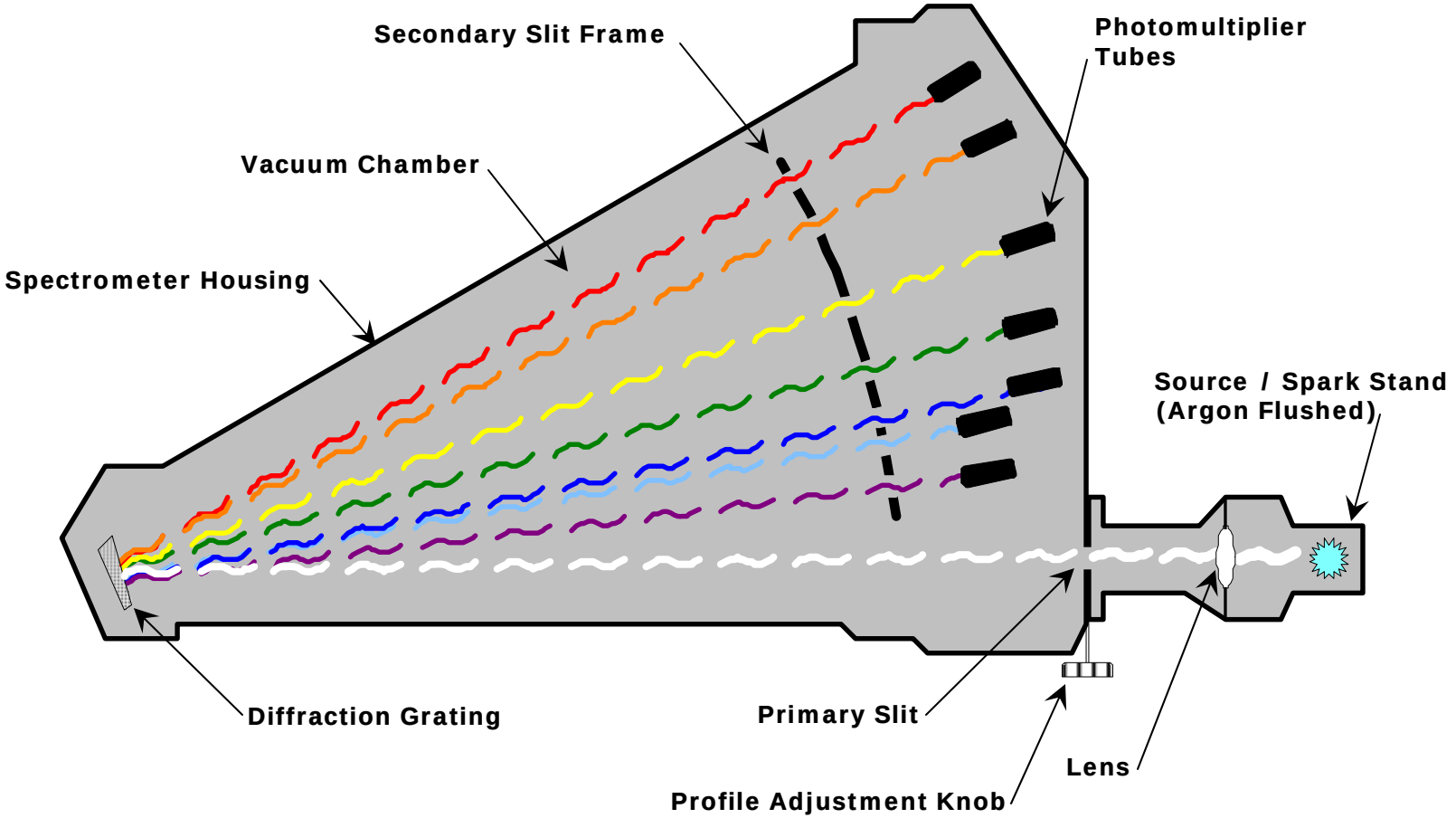


The example here is Mg segregation in 5182 alloy. Segregation occurs for all elements to some degree in all alloys. Mg segregation in 5182 is very drastic thus making a good example. Machining from the spure side of the sample has been found to be best. Consistent depth of cut to the correct depth is critical for obtaining representative chemistry of the melt.

Chemical Analysis

"The Quality of an Analysis is Never Better than the Quality of the Sample"

Spectrometer Schematic



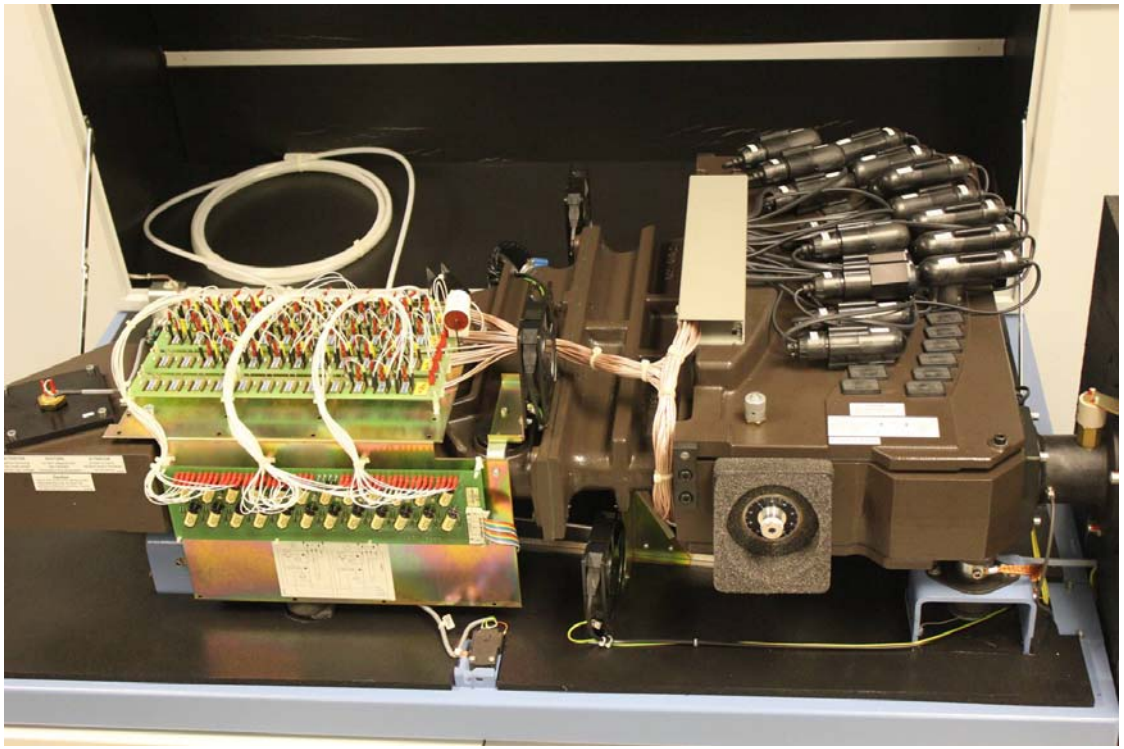
How a Spectrometer Works

- A spark is generated between an aluminum sample and an electrode. It is similar to an arc weld spark.
- This spark produces “white light”. This white light is made up of many colors or wavelengths.
- The light from the spark passes through a lens and then through a primary slit. This lens and slit focus and direct the most intense light on the diffraction grating (like a prism).
- The “profile knob” adjusts the primary slit so the most intense or bright light hits the diffraction grating.
- The “white light” is separated into different colors or wavelengths after it hits the diffraction grating. The diffraction grating behaves like a prism. An example we see is after a rain when the white light of the sun is broken into different colors in water droplets … this is were a rainbow comes from. For aluminum, each different color represents a different element in the sample!
- The separated light then passes through secondary slits and is directed by mirror into a photomultiplier tube. A photomultiplier tube measures the intensity of each color light.

How a Spectrometer Works

• When light hits the photomultiplier tube a current is generated and measured buy the instrument.

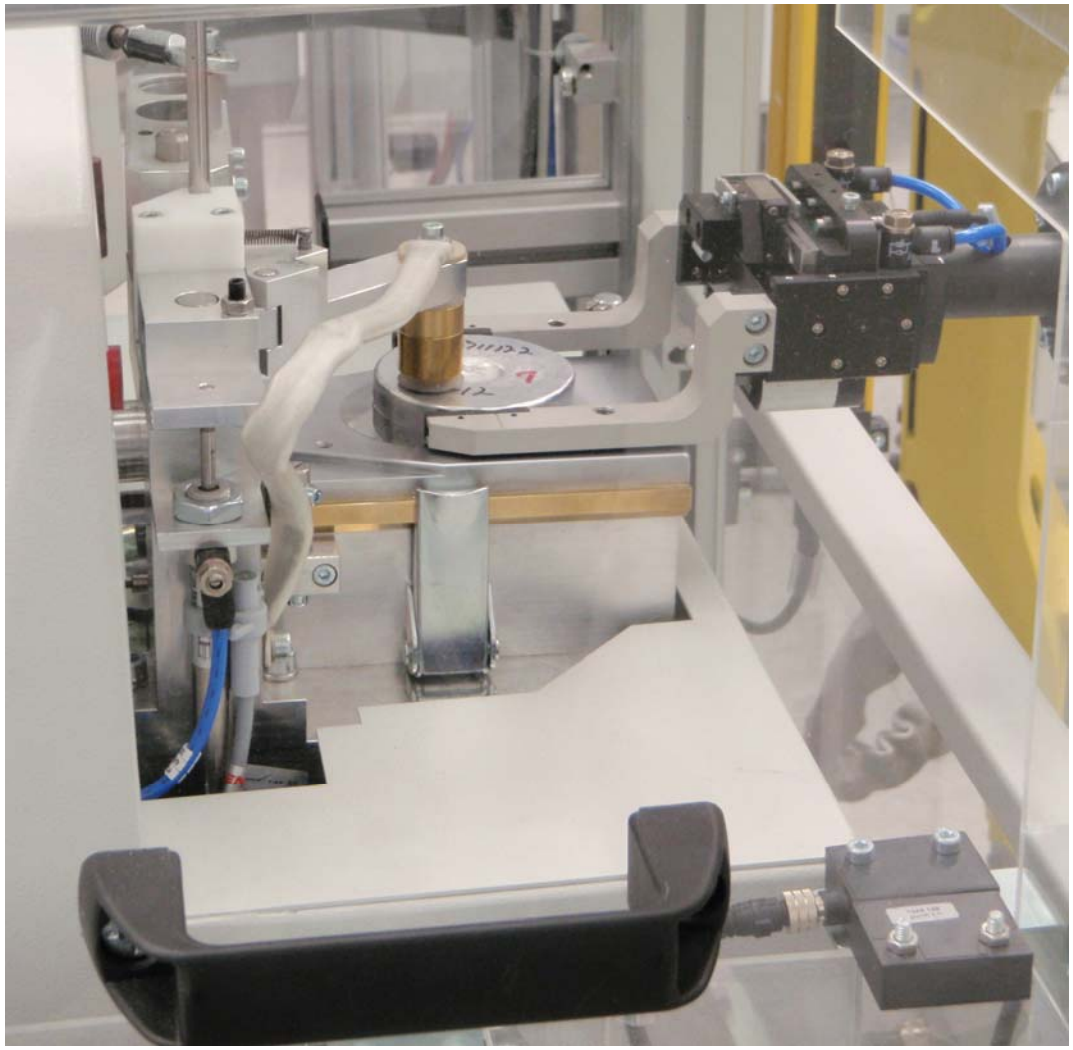
•By developing a calibration curve from known standards with the use of a computer we are able to convert the current measured by the instrument into a weight percent of an element in a sample.



Automated OE Laboratory

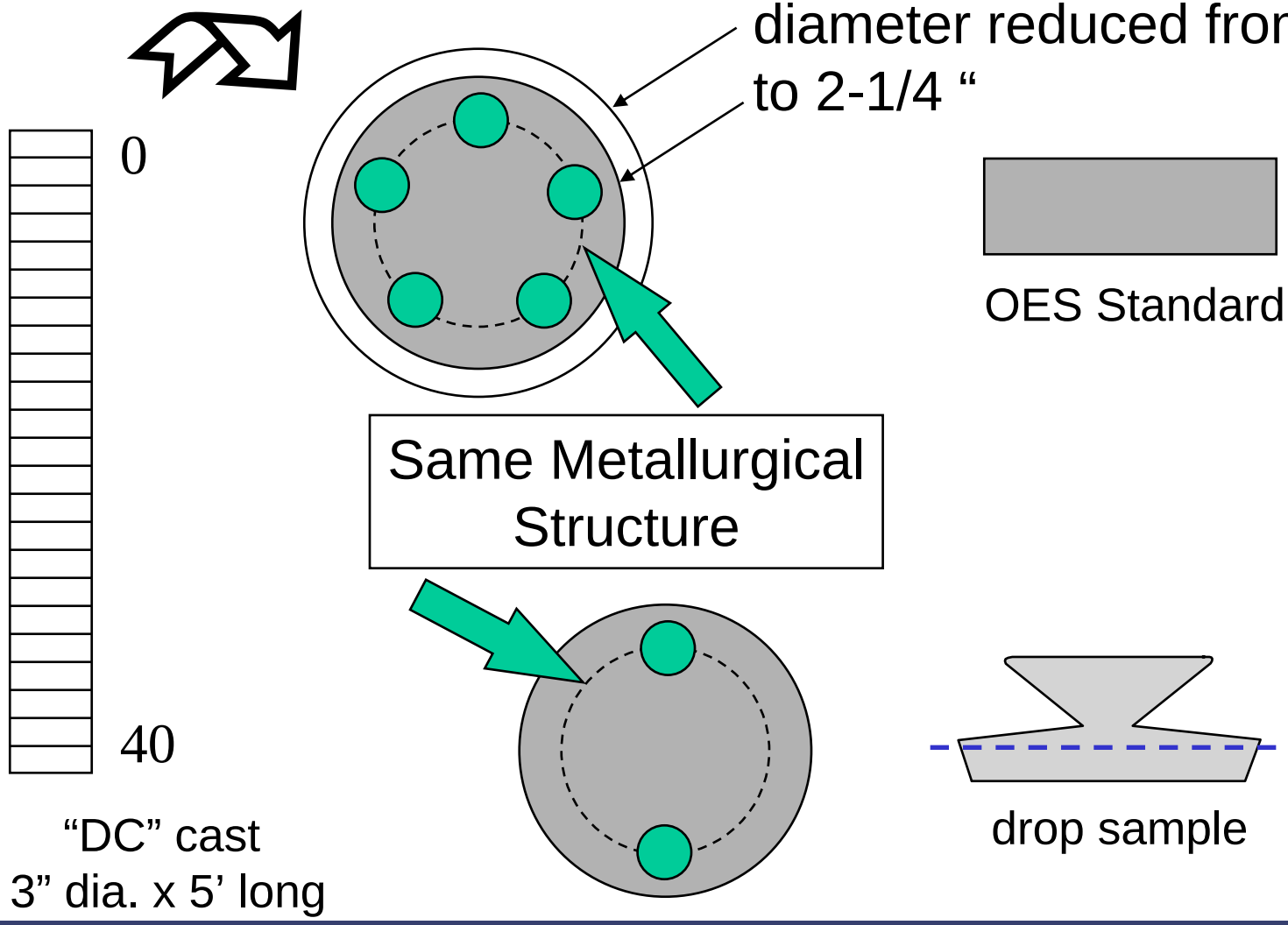
RSI samples are fed into the automated laboratory by the spiral magazine. Samples are automatically analyzed and then sorted.

Automated OE Laboratory



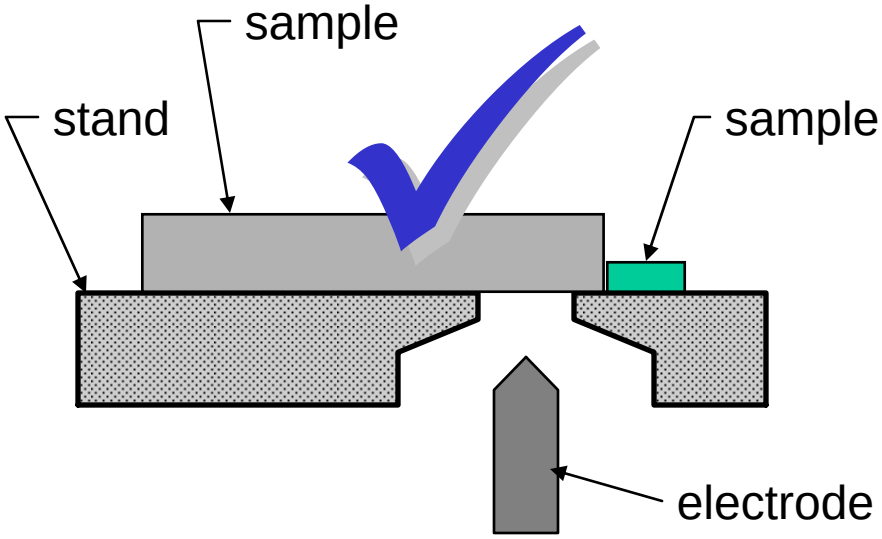
Automated OE Laboratory

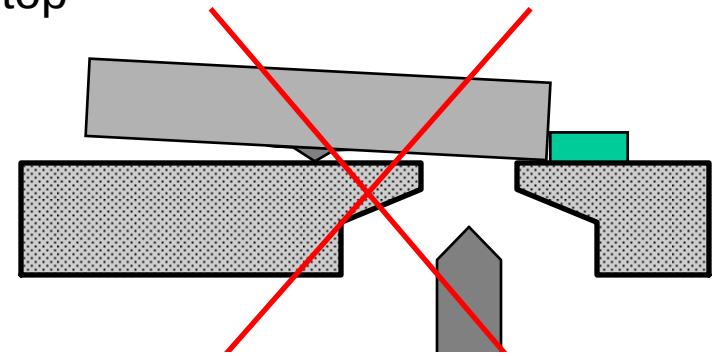
OES Standard Preparation



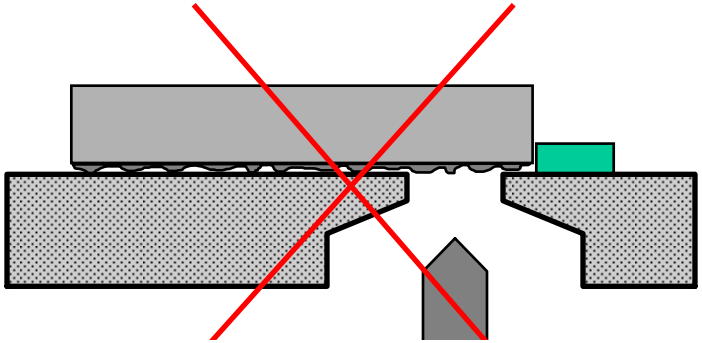
Sample Preparation & Sparking OES analysis
When air is allowed to enter spark stand bad burns result

When using the old quantometer

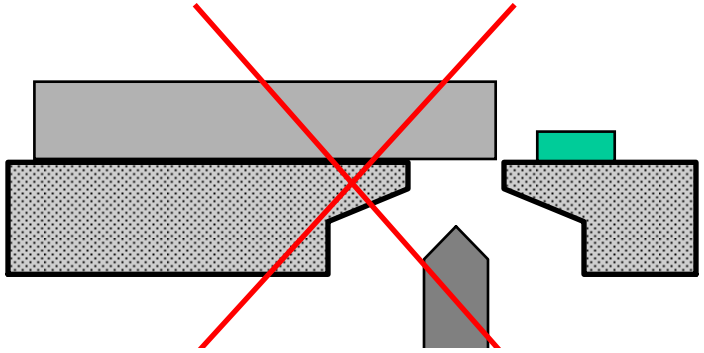




Sample not sitting flat on stand



Rough sample surface



Sample not covering spark hole

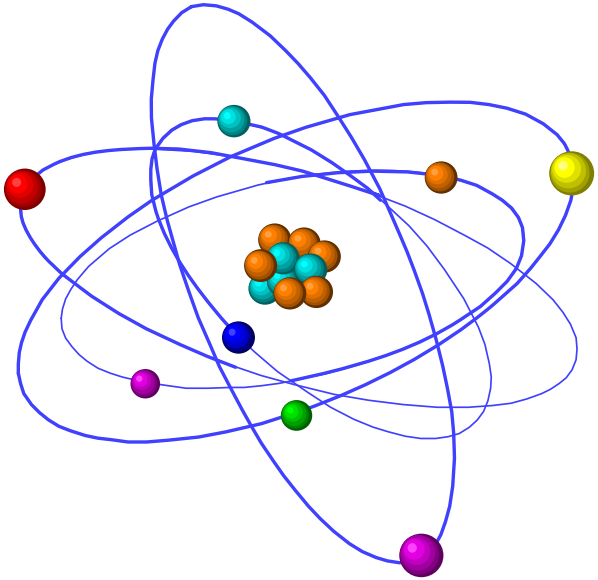
Chemical Sampling Notes

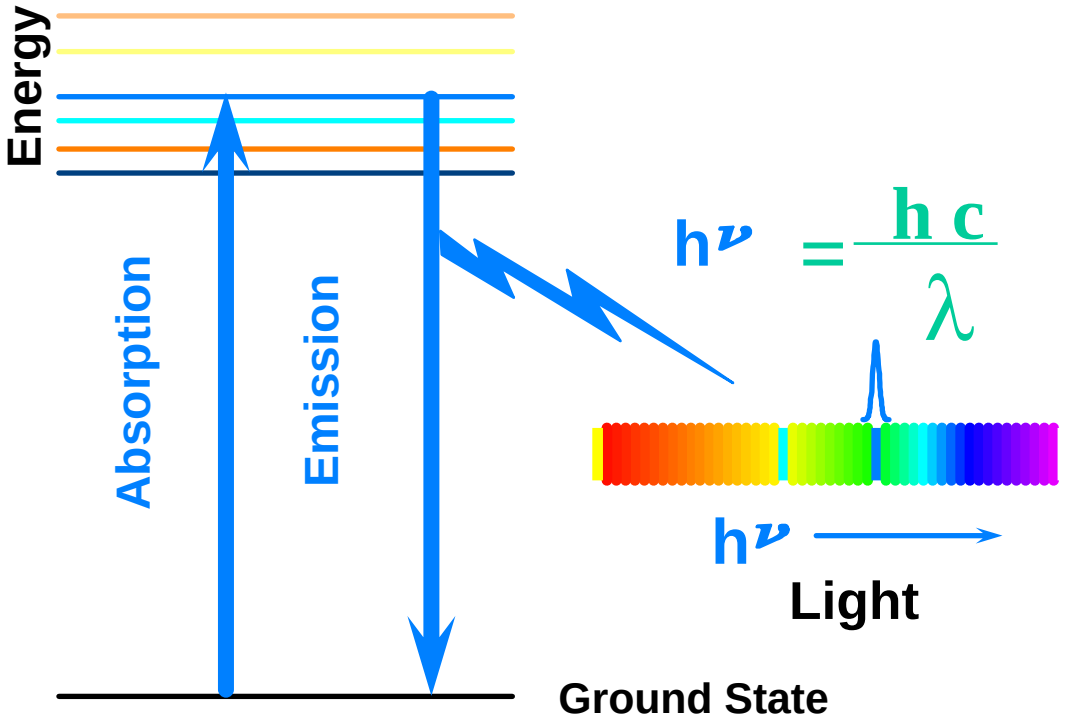
- ✓ Wear all required PPE
- ✓ Inspect sample mold and verify that it is clean, dry and in good condition. Replace if necessary.
- ✓ Preheat, coat and dry sample ladle
- ✓ Allow the furnace to settle if necessary
- ✓ With minimum disturbance push dross away from sample point
- ✓ Carefully submerge sample ladle to the required depth with minimum splashing
- ✓ Lift ladle up and out of metal and pour smoothly and continuously into sample mold. The sprue cavity should be 3/4 full.
- ✓ Discard first sample as a preheat sample

Chemical Sampling Notes, continues

- ✓ Once a sample has completely solidified remove it with a pair of tongs and quench it in a pail of clean water
- ✓ Never break the sprue off before complete solidification
- ✓ Never shake or drop the sample mold
- ✓ At the end of machining the sample must have uniform thickness.
- ✓ The surface must be flat without visible grooves.
- ✓ Prevent the contamination of the surface during machining and handling.
- ✓ Avoid machining the standard or production sample hours before analysis.
- ✓ Reject any malformed sample. Reject any sample with porosity or dross inclusions.

Electronic States of an Atom

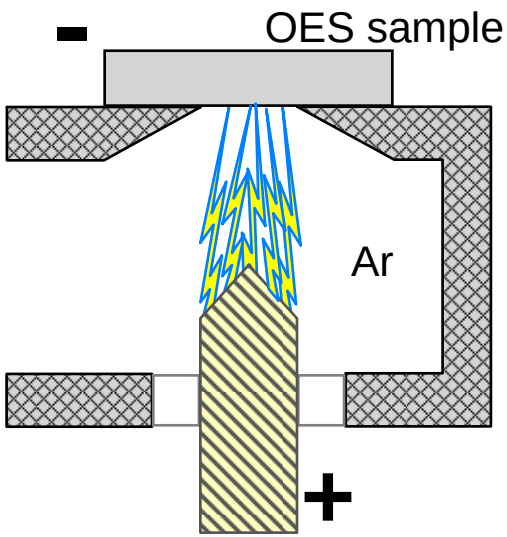


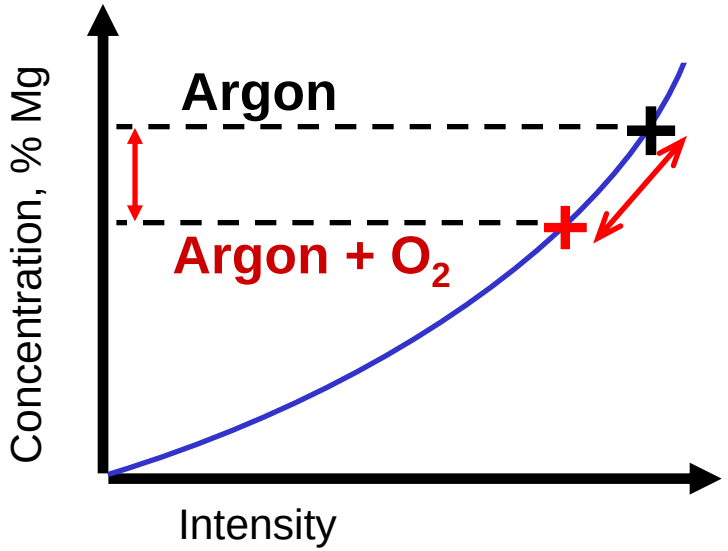


Excited state energies are different for each atom

Effect of Oxygen on OES analysis

(sparking under an Ar atmosphere)





- **contaminated Ar (Argon)**
- **leak in spark stand**
- **oxides/oxygen in dross inside a chemical sample**