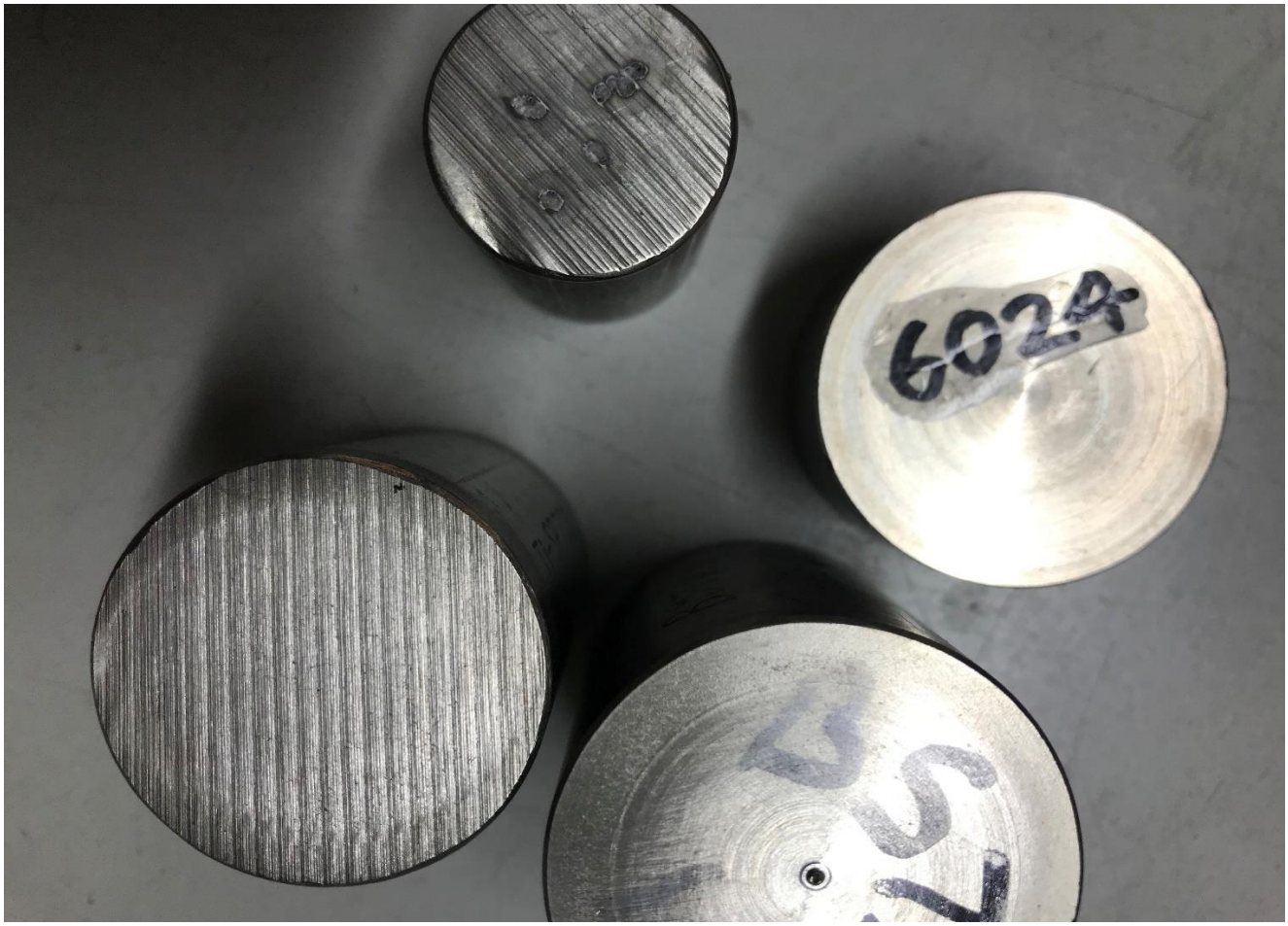
Sample Preparation



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Correct sample preparation is necessary in any chemical analysis to achieve precise and correct results. The purity of the sample should be ensured before taking a measurement to obtain the optimum results when using any elemental alloy analyser. It is important to correctly prepare the surface of the sample before testing. Some technologies are more tolerant to sample surface conditions compared to others.

The extent to which an analyser can tolerate dirty surfaces depends on different variables such as the type of the contamination on the sample surface, the penetration depth of the excitation, analysis spot size, and the nature of physical phenomenon behind the technology.
X-ray fluorescence (XRF), optical emission spectroscopy (OES), or laser-induced breakdown spectroscopy (LIBS) are the most common techniques to perform elemental alloy analysis. The sample preparation required in each of this technology is different. While XRF can tolerate even very dirty sample surfaces, spark OES requires a proper sample preparation to obtain quality results. In the LIBS analysis method, penetration of the laser beam is only a couple of microns deeps, therefore the sample surface should be relatively clean to obtain precise results.

Surface of the Samples

On some of the materials, like stainless steel, nickel, magnesium or titanium alloys, the clear patina or thick oxide layer is not formed on the sample surface. However, samples like copper-based alloys, manganese steels and low alloys steels can create a heavy patina or thick rust which will significantly affects the results of the analysis. Any coating, lacquer or paint must be removed from the sample before analysis. We recommend cleaning the surface of the sample before every analysis to obtain the most accurate results.

Sample Analysis

- When the sample is sparked, approximately 5mg of material is vaporised during the integration of the analytical cycle.

- The analytical result refers only to this part and therefore the structural quality of the sample is extremely important for obtaining accurate and reproducible results.
- A flat surface must be prepared on a sample to provide a good argon seal on the spark stand.

- The most common sample preparation machines are the lathe (for soft metals) and the disc sander (for hard metals).

- The flattened section of the sample must have a homogeneous structure and be free from physical defect such as porosity and shrinkage cracks.

- When the sample is cast and removed from the mould, the outer surface will naturally have an oxidised skin. This must be removed to expose the surface suitable to be analysed and then final preparation can take place.

- During surface preparation the sample must not be overheated otherwise the defects such as surface cracking, oxidation and changes in microstructure will occur

Choosing the Right Sample Preparation Tools

1. Disc Sander/ Linisher

- This surface preparation method is the quickest way to prepare hard metal samples like ferrous, nickel, cobalt, etc...
- The sample must be thick enough so that it can be held with fingers without the risk of injury.
- The aluminium oxide abrasive paper disk must be used.

2. Grinding Machine

- This surface preparation method is suitable to prepare hard metal samples like ferrous, nickel, cobalt etc...
- The magnetic samples will be kept still by a magnetic chuck.
- Non-magnetic samples must be fixed on manual chuck.
- The grindstones must be regularly sharpened.

Manual swing grinder

Grinding wheel

Manual chuck

Magnetic chuck

3. Lathe

- The lathe is used for preparing all soft metals and steel for example lead, aluminium, zinc, and magnesium.
- It is important to keep the cutting tip sharp and clean.
- Different cutting tips must be used for different metals to avoid contamination.