Metal Purity Testing

R. Gowthami, M.E – Embedded System Technologies/ECE, Sri Sairam Engineering College, Chennai, India gowth1207@gmail.com

A. Kumaran, Assistant Professor/ECE, Sri Sairam Engineering College, Chennai, India kumaran.ece@sairam.edu.in

Abstract: There is an increasing demand in precious metal like gold, diamond since they are expensive and cannot be created. Precious metal like gold are tested using acids for their purity which may lead to dissolving of gold in the acid. Hence to avoid corrosion of metal X-ray fluorescence analysis is proposed. The objective of the proposed method is to find the percentage of all the elements present in the any metal. Here X-rays are directed to precious metals (gold) without harming the metal in any way, the x-rays cause them to glow (fluoresce), like in a fluorescent light bulb. With advanced computer detection technology, the machine can separate the colors of the fluorescence into an analysis of the metal. Here the technique used is XRF (X-ray Fluorescence) where x-rays from a sealed tube are used to produce x-rays by secondary fluorescence in samples of interest. The Secondary target fluoresces and excites the sample. The detector detects x-rays from the sample. Graph is plotted using processing techniques and the elements are detected.

I. INTRODUCTION

It is important to test the purity of metal. Nondestructive testing involves the interaction of energy with a material. NDT often involves detecting manufacturing defects and to find the percentage of elements in the metal and, therefore, it is necessary to understand how defects and damage occur. A fine finished product can be a precious metal or it contains other materials/impurities. Carat is the unit for measuring the purity of material. Although it is almost certainly impossible to remove every single impurity, fine metal is 99.99% pure. Carat age of a metal is calculated in percentage. Although it is displayed and referred to in its decimal value and / or out of a thousand parts. An example in silver most commonly used is known as 925 and is actually 92.5% fine silver and 7.5% alloy metals. In the past metal are tested for their purity using acids such as HCl (Hydro Chloric acid) and Nitric Acid which may damage the metal content. Acid test was replaced by electronic testers which also had several disadvantages. Hence here nondestructive testing is applied so that percentage of metal are accurately tested and destruction is reduced.

II. PRINCIPLE

X-Ray Fluorescence (XRF) is a very simple analytical technique. X-rays excite atoms in a sample, which emit X-rays at energies characteristic of each element. A detector measures the energies and intensities of the emitted X-rays. From this, one deduces which elements are present and their concentrations.

Setting up a system for first order results is straightforward. One can easily identify the major constituents in simple samples and obtain approximate concentrations. This is sufficient for some applications. However, second order effects are quite important for many applications. To obtain high accuracy, identify constituents with low concentrations, or operate on a wide variety of samples, second order effects must be addressed. To remove these second order effects and obtain high accuracy, properly configuring both the hardware and software is critical. Understanding the physical mechanisms responsible for these effects is helpful when one is configuring a system to obtain accurate results.

The principle of Metal purity testing is as follows: Photons from the source irradiate the sample. Shown here is an X-ray tube with Ag anode. Atoms in the sample are excited, then de-excite by emission of X-rays at energies characteristic of the elements in the sample. The intensities are related to the concentration of each element in the sample.
sample and to the excitation probabilities. The characteristic X-rays interact in the detector. Each X-ray releases a pulse of charge, proportional to the energy deposited. The signal processing electronics measures the total charge (i.e. total energy) of each pulse that is of each individual X-ray. The signal processing electronics sum the results of many interactions into a histogram, showing the number of events in each of many energy bins. This is the energy spectrum. The peaks in the spectrum correspond to the elements in the sample. The number of X-rays in each peak is proportional to the number of atoms. Analysis software detects which peaks are present, finds the intensity of each peak, and computes the concentration of each element in the sample.

IV. HOW DOES THE ANALYSIS SOFTWARE WORK?

The matlab software uses the measured spectrum to deduce the concentration of elements in the spectrum. There are three major steps to the analysis: The measured spectrum produced by the software is as shown in Fig: 1 Unravel the detector response to recover the incident photo peaks. The output of this step is a processed spectrum, ideally showing only the incident photo peaks. 

[1] Deconvolve the photo peaks to determine the intensity of the X-rays interacting in the detector. The output of this step is a table of the intensities in each photo peak to be analyzed.

[2] Account for attenuation and matrix effects to determine the concentrations of the elements in the sample. The output of this step is a table of concentrations, as in Table 1.

A. MATLAB STEP BY STEP

The steps by which matlab goes from the raw spectrum to the final result contains more steps. Before one can analyze a sample, there are several key actions required. They include (1) setting up the hardware, (2) launching and configuring the software, (3) performing an energy calibration, (4) calibrating the FP software using reference materials, and (5) acquiring the spectrum to be analyzed. The spectrum acquired in matlab is the plot of elements which may be present in the material. After the spectrum is plotted, the spectrum should be processed to get the percentage of each elements in the metal.

A. Unraveling detector response

Smooth fluctuations: The first step in processing is to apply a filter which smooths the spectrum. This reduces the effects of counting statistics and will improve the ability of the software to recognize the background and to remove the true peaks from background.

Correct for escape peaks: The software applies known corrections to remove the escape peaks and put the counts into the photo peaks. This removes possible interferences and corrects the photo peak net area for escapes. These false peaks have been removed and their counts added back into the photo peaks.

Remove sum peak:The software applies known corrections to remove the sum peaks and put the counts into the photo peaks. This removes possible interferences and corrects the photo peak net area.

ii. Remove continuum

Background removal:The first background removal step applies what is essentially a low pass filter to estimate the background, using a proprietary function to model the slowly changing background. Without attempting to separate the distinct physical mechanisms, the background is removed. The smoothly varying continuum should be removed.

Blank subtraction:Blank subtraction removes artifacts which do not vary significantly from one spectrum to another and cannot be handled with other processing.

Compton peak removal: Compton peak processing estimates the intensity of the Compton and Rayleigh scatter peaks. This is used during some of the deconvolution and quantitative analysis methods, depending on the options chosen. The spectrum is not shown here since this does not appear as a change in the spectrum. This processing step produces certain parameters used in deconvolution.

1)iii. Deconvolution
Deconvolution is the process of assigning the areas to the peaks of interest. The output of this step is a list of “elemental intensities”. This is depicted in the spectrum display as the Gaussian peaks, with the appropriate areas and centroids. The results of the deconvolution for the spectrum shows the intensities with respect to area, height and width as shown in fig 2.

![Fig 2: Process of deconvolution to find peaks](image)

**B. Quantitative Analysis**

This is the step which relates the net intensity of each peak to the concentration in the sample. This processing includes attenuation along with matrix effects, absorption and enhancement in the sample. The result of the quantitative analysis step is not shown in the spectrum, but in the output table, such as in Table 1. In the table the intensities of the elements are plotted based on their percentage.

<table>
<thead>
<tr>
<th>Table 1: Percentage of elements based on intensities</th>
</tr>
</thead>
</table>

**REFERENCES**

[1] Johanna Nelson¹, Xiaojing Huang², Jan Steinbrener², Shapiro: ‘High-resolution x-ray diffraction microscopy of specifically labeled yeast cells’ PNAS, Edited by Margaret M. Murnane, University of Colorado at Boulder, Boulder, CO, and approved March 11, 2010


