

2. Experimental Methodology

We collected various Industrial Application Steels Viz., Plain & Medium Carbon Steel, Low Carbon Steel, Bearing Steel, Low & Medium Alloy Steel, Platinum Tool Steel and Ferritic Stainless Steel for the study; the steel rod of raw material cut from abrasive cutting Machine and made Diameter of $\phi 20$ mm to $\phi 22$ mm and the thickness is 10-15 mm. All the samples are investigated by Wet Chemistry and Optical Emission Spectra and Positive Material Identification.

2.1 Wet Chemistry

These methods include gravimetric and titrimetric techniques. An example of a gravimetric technique is the precipitation of chloride ion with silver to form a silver chloride precipitate which is dried and weighed to determine the chloride concentration in the original sample solution. Titrimetric procedures are typically based on acid-base reactions or complexing agents for metal ions. Since wet chemical analyses are now less common for the analysis of metals and similar inorganic materials, the remainder of this section will focus on the instrumental methods of analysis [4, 5]. For this wet analysis we used the following standard procedure to determine the chemistry of steel i.e. assigned

elements, IS 228 Part 1 to 9 and ASTM E 353 [6,7,8,9 10 & 11].

2.2 Optical Emission Spectra

Optical emission spectrometry involves applying electrical energy in the form of spark generated between an electrode and a metal sample, whereby the vaporized atoms are brought to a high energy state within a so-called "discharge plasma". These excited atoms and ions in the discharge plasma create a unique emission spectrum specific to each element. Thus, a single element generates numerous characteristic emission spectral lines. Therefore, the light generated by the discharge can be said to be a collection of the spectral lines generated by the elements in the sample. This light is split by a diffraction grating to extract the emission spectrum for the target elements. The intensity of each emission spectrum depends on the concentration of the element in the sample. Detectors (photomultiplier tubes) measure the presence or absence or presence of the spectrum extracted for each element and the intensity of the spectrum to perform qualitative and quantitative analysis of the elements [12, 13]. For this OES Study, the sample to be free from oil, rust and contaminations and also required very fine polished surface so we used milling machine to prepare the sample for this Investigations.

Table 3.1: Test Results of Chosen samples by Wet Chemistry Method

| S. No | Sample ID Code & Size | Spec. | Observed Chemistry (%) | | | | | | | | | | Observed Grade | |
|-------|-------------------------------|--------------------|------------------------|------|------|------|----|----|----|---|------|-------|----------------|-------------------|
| | | | Mn | Mo | Cr | Ni | Cu | Pb | Fe | C | S | P | | |
| 1 | P1 ($\phi 20$ mm x10 mm) | IS 2062 Grade A | 0.22 | Tr | Tr | Tr | Tr | Tr | Tr | R | 0.23 | 0.045 | 0.04 | IS2062 A Grade |
| 2 | P2 ($\phi 21$ mm x10 mm) | AISI 1010 | 0.32 | Tr | Tr | Tr | Tr | Tr | Tr | R | 0.08 | 0.037 | 0.03 | AISI 1010, B |
| 3 | P3 ($\phi 21$ mm x10 mm) | AISI E 52100 | 0.45 | Tr | 0.9 | Tr | Tr | Tr | Tr | R | 0.98 | 0.02 | 0.02 | AISI E2100 |
| 4 | P4 ($\phi 20$ mm x 10 mm) | C10XX | 0.86 | Tr | Tr | Tr | Tr | Tr | Tr | R | 0.07 | 0.03 | 0.03 | IS 531 /C10xx |
| 5 | P5 ($\phi 22$ mm x 10 mm) | O-1, 0-6 | 1.55 | 0.1 | 0.20 | Tr | Tr | Tr | Tr | R | 1.25 | 0.03 | 0.02 | O6 |
| 6 | P7 ($\phi 20$ mm x 10 mm) | T5 | 0.30 | 1.29 | 3.75 | Tr | Tr | Tr | Tr | R | 0.80 | 0.02 | 0.02 | T5 |
| 7 | P8 ($\phi 21$ mm x 10 mm) | AISI 422 | 0.90 | 0.70 | 11.5 | 0.2 | Tr | Tr | Tr | R | 0.20 | 0.025 | 0.02 | SS 422 |
| 8 | P9 ($\phi 21$ mm x 10 mm) | AISI 431 | 0.60 | Tr | 16.7 | 1.4 | Tr | Tr | Tr | R | 0.18 | 0.035 | 0.03 | SS 431 |
| 9 | P10 ($\phi 22$ mm x10 mm) | 2 ¼ Cr | 0.50 | 0.10 | 2.20 | 0.12 | Tr | Tr | Tr | R | 0.14 | 0.01 | 0.01 | 2 1/4Cr |

Note: Tr- Trace (i.e. less than 0.02%)

R- Reminding Major Assignment in % (Base element)

2.3 XRF Spectra (PMI)

The XRF principle (X- Ray Fluorescence) is one of the methods for PMI. Equipment used contains low radioactive sources (isotopes) or x-ray tubes. The exposed material reflects the radiation, generating energy. As every element has its own atomic structure, this reflection will generate a different energy level for every element. This energy is measured and detected, thus identifying the alloy elements. However, it is important that the surface is identical to rest

of the material. Oxides, coatings and dirt on the material will influence the identification results. Also the surface must be smooth. Interesting to know is that the exposure of radiation is sufficiently low, so that extra safety measures are not required [13]. We pre-cleaned the surface by fine grid emery sheets for free from oil, rust and contaminations to this PMI Study.



Figure 2.3: (a) PMI Equipment Used for this Study

P7,P8 and P9 as per the reference Standards and determined Procedures. The results of assigned major elements and respect to the grade are shown in Table 3.1. Using Wet Method we confirmed the grade of Steel and the results once again verified by Optical emission Spectra then Proceeded for Positive Material Identifications.

3.2 Optical Emission Spectra

The Optical Emission Spectrum was done for the chosen samples Viz., P1, P2, P3, P4, P5, P7,P8 and P9 at M/s.Vibrant NDT Services Pvt.Ltd.,Chennai, as per the reference Standards and-determined Procedures. The results of assigned major elements and respect to the grade are shown in Table 3.2.

3. Results and Discussions

3.1 Wet Chemistry

Wet Analysis done at M/s.Vibrant NDT Services Pvt. Ltd., Chennai. for the chosen samples Viz., P1, P2, P3, P4, P5,

Table 3.2: Test Results of Chosen samples by Optical Emission Spectra

| S. No | Sample ID Code & Size | Spec. | Observed Chemistry (%) | | | | | | | | | | Observed Grade |
|-------|-------------------------|--------------------|------------------------|------|------|------|------|----|----|------|-------|------|--------------------|
| | | | Mn | Mo | Cr | Ni | Cu | Pb | Fe | C | S | P | |
| 1 | P1 (ø20 mm x10 mm) | IS 2062 Grade A | 0.25 | 0.01 | 0.02 | 0.01 | 0.02 | - | R | 0.21 | 0.045 | 0.04 | IS2062' A'Grade |
| 2 | P2 (ø21 mm x10 mm) | AISI 1010 | 0.32 | 0.01 | 0.0 | 0.02 | 0.02 | - | R | 0.07 | 0.037 | 0.03 | AISI 1010, B |
| 3 | P3 (ø21 mm x10 mm) | AISI E 52100 | 0.69 | 0.01 | 0.9 | 0.02 | 0.09 | - | R | 0.99 | 0.02 | 0.02 | AISI E2100 |
| 4 | P4 (ø20 mm x 10 mm) | C10XX | 0.90 | 0.01 | 0.0 | 0.01 | 0.0 | - | R | 0.06 | 0.03 | 0.03 | IS 531 /C10xx |
| 5 | P5 (ø22 mm.x 10 mm) | O-1, 0-6 | 1.10 | 0.1 | 0.20 | 0.0 | 0.05 | - | R | 1.21 | 0.03 | 0.02 | O6 |
| 6 | P7 (ø20 mm x 10 mm) | T5 | 0.40 | 1.25 | 3.90 | 0.04 | 0.09 | - | R | 0.84 | 0.02 | 0.02 | T5 |
| 7 | P8 (ø21 mm x 10 mm) | AISI 422 | 0.90 | 0.75 | 11.5 | 0.9 | 0.04 | - | R | 0.18 | 0.025 | 0.02 | SS 422 |
| 8 | P9 (ø21 mm x 10 mm) | AISI 431 | 0.64 | 0.15 | 2.30 | 1.9 | 0.04 | - | R | 0.16 | 0.035 | 0.03 | SS 431 |
| 9 | P10 (ø22 mm x 10 mm) | 2 ¼ Cr | 0.50 | 0.90 | 2.20 | 0.16 | 0.08 | - | R | 0.14 | 0.01 | 0.01 | 2 1/4Cr |

R- Reminding Major Assignment in % (Base element)

3.3 XRF Spectra -PMI

The X-Ray Fluorescence Spectrum was done for the chosen samples Viz., P1, P2, P3, P4, P5, P7,P8 and P9 at M/s.Vibrant NDT Services Pvt.Ltd., Chennai, as per the

reference Standards and determined Procedures. The XRF Spectra and the observed Results are given below,

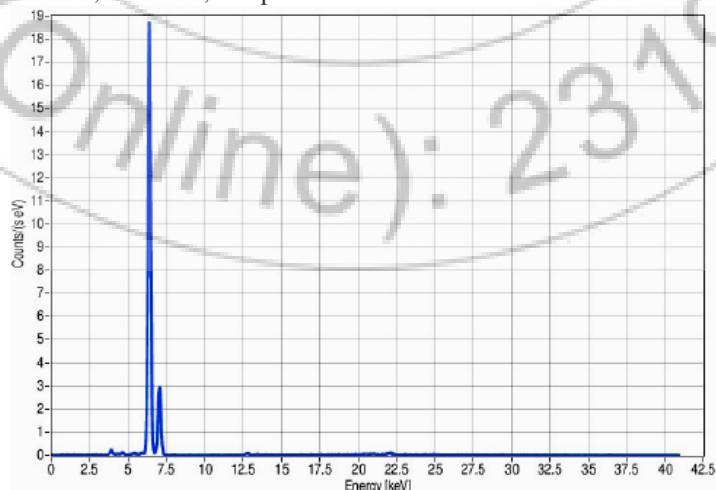


Figure 3.3 (a) XRF Spectra for Plain Carbon Steel

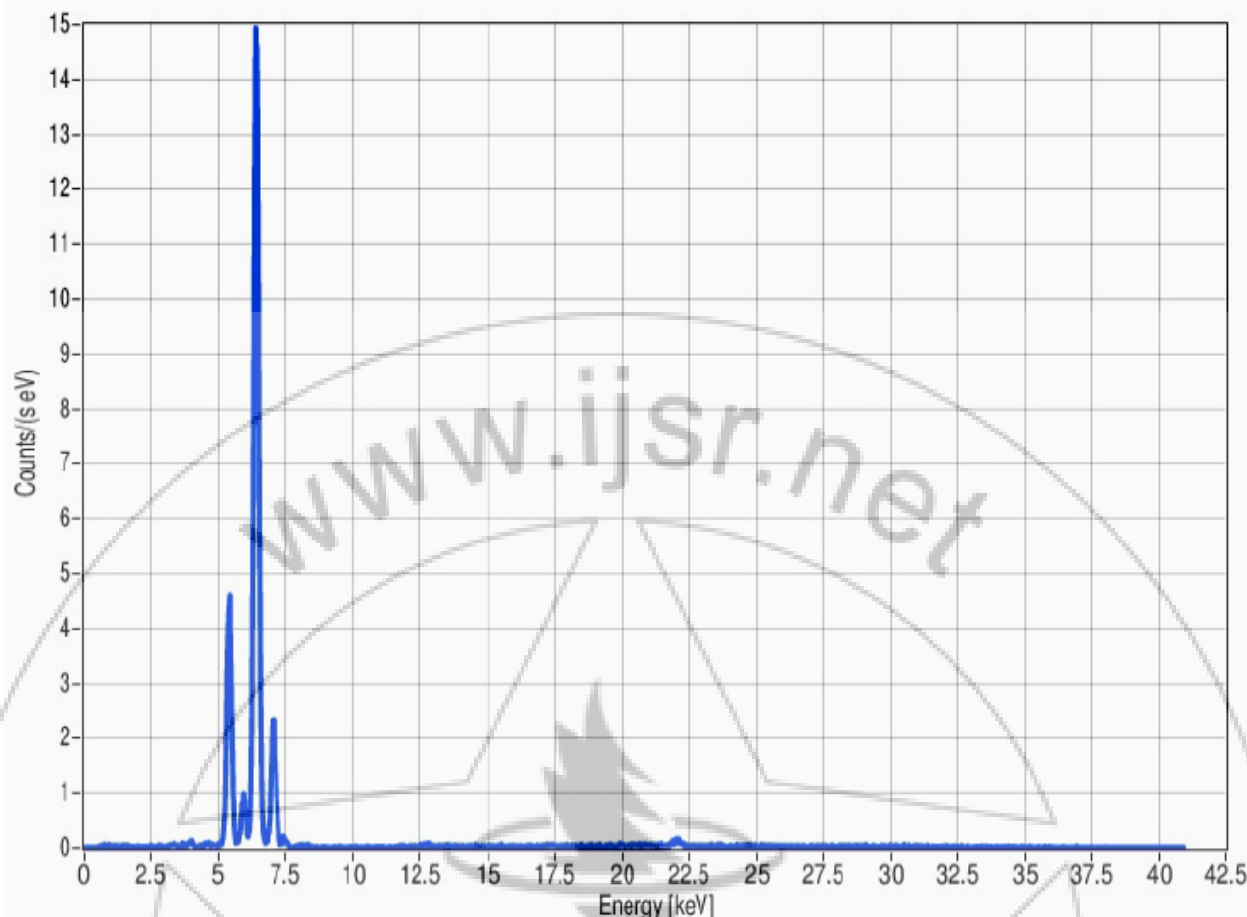


Figure 3.3 (b): XRF Spectra for Stainless Steel

3.4 Correlation of Obtained Results from XRF Spectra, OES & Wet Chemistry

In the case of XRF Spectra does not shown Percentage of Carbon because it has used as a reference source for diffraction of X-ray so we consider the correlations except the Carbon percentages and the results of correlations are found satisfactory with respect to the percentages of major assigning elements.

4. Conclusion

From the Study of XRF Spectra, It has given best results for Alloy Steels without destruction of samples and Percentages of Positive Materials vis., Cr, Ni, Mo, Co, W, Mn, V, T, Cu, Al, etc., and also it has given authenticate results of grade Confirmations of this work i.e. chosen Steel Samples namely P1, P2, P3, P4, P5, P7, P8, P9 and P10. Wet Chemistry Method and Optical Emission Spectra are proven techniques but the samples are destructed and the percentages of assigning elements not repeatable for example in OES techniques little bit variations found due to the sitting position of sample during arc/ spark formations. Similarly we found variations during analysis of wet method by human errors like weighting of samples for analysis and temperature factors, used chemicals like that so the measurement of uncertainty is very difficult in both Wet and OES Techniques. From the overall Investigations, We conclude that Positive Material Identification (PMI) i.e. XRF Spectra is the only best technology for Qualitative

Chemical Analysis of Ferrous and Non-Ferrous Components by Non Destructive Evaluations but It will support only the positive /Alloy elements not for Carbon percentage and Negative element like S, P, Si, etc.,

References

- [1] N.G. Farzane, L.V. Ilyasov, A.Yu. Azim-Zade, Technological measurements and devices, Visshaya Shkola, Moscow, 1989, 456 p (in Russian).
- [2] I.P. Alimarin, V.M. Ivanov, Practical handbook on physical-chemical methods of analysis, Izd.MGU, Moscow, 203 p (in Russian).
- [3] Andriy GRYTSKIV, Yaroslav GRYTSKIV, Anatoliy TROFYMCHUK, determination of the Composition of steels by inductively coupled plasma optical emission spectrometry (icp-oes) using the thermo scientific icap 6500 duo spectrometer, **Chem. Met. Alloys 2 (2009) 177-182.**
- [4] L.V. Vilkov, Yu.A. Pentin, Physical methods of investigation in chemistry. Structural methods and optical spetroscopy, Visshaya Shkola, Moscow, 1987, 366 p (in Russian).
- [5] William D. Callister, Jr, Materials Science and Engineering – An introduction, sixth edition, **John Wiley & Sons, Inc. 2004.**
- [6] **IS 228, Part 1** Determination of Carbon Percentage in Low Alloy Steel, **2005.**
- [7] **IS 228, Part 2** Determination of Manganese Percentage in Low Alloy Steel, **2005.**

- [8] **IS 228**, Part 5 Determination of Nickel Percentage in Low Alloy Steel, **2005**.
- [9] **IS 228**, Part 6 Determination of Chromium Percentage in Low Alloy Steel, **2005**.
- [10] **IS 228**, Part 7 Determination of Molybdenum Percentage in Low Alloy Steel, **2005**.
- [11] **ASTM E353, Sec.172**, Determination of Nickel Percentage in Stainless Steel, **2009**.
- [12] **ASTM E353, Sec.212**, Determination of Chromium Percentage in Stainless Steel, **2009**.
- [13] **ASTM E1476, Standard** guide for Metal Identification, Grade Verfications, and Sorting 2009.

Author Profile



P. Kuppuraj received the B.Sc. and M.Sc. degrees in Physics from University of Madras in 2005 and 2007, respectively. During Nov.2007- Sep.2011, he stayed in M/s. Aero Laboratories, Chennai, India as Quality Manager of Posts and the same year of sep. 2011, he shifted to M/s. Roop Telsonic Ultrasonic Ltd., as a Sr. Engineer from Sep.2011 to May 2012. He now with M/s.Vibrant NDT Services Pvt.Ltd., Chennai, India as a Sr. Manager –Development from May 2012 and also he is pursuing his Research work in physics by Part Time at PG & Research Department of Physics, Pachiyappa’s College under the University of Madras from August 2012.