Determine the Chemistry of Selected Steel by NDE Technique of Positive Material Identification (PMI)

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Abstract: A great number of procedures have been developed and today a wide assortment of special analytical equipment for the analysis of chemical compositions of metal and alloys, including those widely spread and industrially produced, exists [1]. However, new analytical tasks for the control of steel products, or for individual parts made from them, constantly appear, and it is sometimes difficult to choose the appropriate method and device for the analysis. Very often complications appear when it is necessary to test a finished product, which must be left in for further use. The analysis must then be performed, or the sample taken, in such a way that the object does not loose its technical assignment or demand future restoration or repair. In addition, the more important and the more expensive the object, the less damage there must be and, at the same time, the more accurate the analysis must be. As a rule, these two conditions contradict each other. The list of objects requiring strict control of their composition is long: boilers, which operate under high pressure, and supplementary equipment; tanks for keeping fuel, lubricants and flammable materials; load-lifting or transportation mechanisms, cranes, and so on [2]. These problems are accelerated to the researchers of Metal chemist and got solutions i.e. Identification of Positive Materials by XRF Techniques of Non Destructive Test and Evaluations (NDE). Often, famously it has called Positive Material Identifications (PMI). It is known that non-destructive control of the composition can be conducted by X-ray analysis and portable compact analyzers have been created for performing analyses under field conditions. Based purely on physical features, X-ray fluorescence analysis is realized in high-quality laboratories. In this present study, we would like to develop a procedure for analyzing some selective industrially used steel products using XRF Spectra i.e. PMI and the same comparatively analyzing by Optical Emission Spectrometer (OES) and Wet Chemistry and also verify the obtained results to prove the best one for NDE.

Keywords: XRF Spectra, PMI, Steel Chemistry by NDE, Positive Material Identification, OES, etc.

1. Introduction

Every Sphere of human activities, we come across several thinks, where there are fruits of problems and solvable research progressing in Metal Chemistry. On today, determination of chemistry of Steel and Its grade confirmation within the stipulated time, is one of the Major difficulties in all type of Industries like Automotives, Railways, Aerospace Components, Pressure Vessels manufactures, and Engineering Constructions, Solar, Thermal, Hydro, Windmill, Nuclear Power Plants, Pharmaceutical, Chemical, Petrochemical, Oil & Gas Refinery Plants, etc., Nowadays, the Quantitative chemical analysis may be performed by one or more complimentary techniques, commonly including spark optical emission spectroscopy (Spark-OES), inductively-coupled plasma spectroscopy optical emission spectroscopy (ICP-OES), xray fluorescence spectroscopy (XRF), wet chemical analyses, combustion methods, and inert gas fusion (IG). The specific technique chosen will depend on the type of sample, quantity of material available for analysis, desired result, and cost constraints. In most cases, the applicable analysis techniques can detect parts-per-million concentrations or better. Most of these techniques are destructive to the original sample. XRF can be performed nondestructively and Spark-OES can be performed with only minimal surface damage if the specimen size configuration allows the part to fit into the instrument without cutting. For the remaining methods discussed here, a small specimen is removed from the sample and is consumed in the analysis. Prior to the widespread availability of analytical instruments, chemical

analyses were performed by dissolving the sample and performing a specific chemical reaction with a standardized reagent for each element of interest. These 'wet chemistry' techniques are typically labor intensive and time consuming, and sometimes less accurate than the current instrumental methods [3].

Steel Industrialist widely used wet chemistry and optical emission spectra techniques for confirmations of their Steel Chemistry, but it was destructive techniques. We know that, the destructive techniques need part or cut position of samples and the tested sample not in use so they were faced many economical difficulties viz., preparation of test samples, timely services and Material Mix-ups after Inspection due to without traceability, etc., and also the destructive techniques not support for hundred percentages of Inspection to the components. These problems are accelerated to the researchers of Metal chemist and got solutions i.e. Positive Material Identification by Non Destructive Testing and Examination (NDT / NDE), which is an indispensable technique. In this present study, we would like to develop a procedure for analyzing some selective industrially used steel products using XRF Spectra i.e. PMI and the same comparatively analyses by Optical Emission Spectrometer (OES) and Wet Chemistry and also verify the obtained results to prove the best one for determine the steel chemistry.

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 ø20 mm to ø 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 complexion 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.

S .	Sample ID Code &	Spec.		Observed Chemistry (%)								Observed	
N٥	Size		Mn	Mo	Cr	Ni	Cu	Pb	Fe	С	S	Р	Grade
1	P1	IS 2062	0.22	Tr	Tr	Tr	Tr	Tr	R	0.23	0.045	0.04	IS2062*
	(ø20 mm x10 mm)	Grade A											A'Grade
2	P2	AISI 1010	0.32	Tr	Tr	Tr	Tr	Tr	R	0.08	0.037	0.03	AISI
	(ø21 mm x10 mm)												1010, B
3	P3	AISI	0.45	Tr	0.9	Tr	Tr	Tr	R	0.98	0.02	0.02	AISI
	(ø21 mm x10 mm)	E 52100											E2100
4	P4	C10XX	0.86	Tr	Tr	Tr	Tr	Tr	R	0.07	0.03	0.03	IS 531
	(ø20 mm x 10 mm)												/C10xx
5	P 5	O-1, 0-6	1.55	0.1	0.20	Tr	Tr	Tr	R	1.25	0.03	0.02	O6
	(ø22 mm x 10 mm)												
6	P 7	T5	0.30	1.29	3.75	Tr	Tr	Tr	R	0.80	0.02	0.02	T5
	(ø20 mm x 10 mm)												
7	P8	AISI 422	0.90	0.70	11.5	0.2	Tr	Tr	R	0.20	0.025	0.02	SS 422
	(ø21 mm x 10 mm)												
8	Р9	AISI 431	0.60	Tr	16.7	1.4	Tr	Tr	R	0.18	0.035	0.03	SS 431
	(ø21 mm x 10 mm)												
9	P10	2 ¼ Cr	0.50	0.10	2.20	0.12	Tr	Tr	R	0.14	0.01	0.01	2 1/4Cr
	(ø22 mm x10 mm)												

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

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

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

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, 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.

Table 3.2. Test Results of Chosen samples by Optical Emission opectra													
S.	Sample ID Code &	Spec.	Observed Chemistry (%)									Observed Grade	
No	Size		Mn	Mo	Cr	Ni	Cu	Pb	Fe	С	S	Р	Grade
1	P1	IS 2062	0.25	0.01	0.02	0.01	0.02	-	R	0.21	0.045	0.04	IS2062'
	(ø20 mm x10 mm)	Grade A											A'Grade
2	P2	AISI 1010	0.32	0.01	0.0	0.02	0.02	-	R	0.07	0.037	0.03	AISI
	(ø21 mm x10 mm)												1010, B
3	P3	AISI	0.69	0.01	0.9	0.02	0.09	-	R	0.99	0.02	0.02	AISI
	(ø21 mm x10 mm)	E 52100											E2100
4	P4	C10XX	0.90	0.01	0.0	0.01	0.0	-	R	0.06	0.03	0.03	IS 531
	(ø20 mm x 10 mm)												/C10xx
5	P5	0-1, 0-6	1.10	0.1	0.20	0.0	0.05	-	R	1.21	0.03	0.02	O6
	(ø22 mm x 10 mm)												
6	P7	T5	0.40	1.25	3.90	0.04	0.09	-	R	0.84	0.02	0.02	T5
	(ø20 mm x 10 mm)												
7	P8	AISI 422	0.90	0.75	11.5	0.9	0.04	-	R	0.18	0.025	0.02	SS 422
	(ø21 mm x 10 mm)												
8	P9	AISI 431	0.64	0.15	2.30	1.9	0.04	-	R	0.16	0.035	0.03	SS 431
	(ø21 mm x 10 mm)												
9	P10	2 ¼ Cr	0.50	0.90	2.20	0.16	0.08	-	R	0.14	0.01	0.01	2 1/4Cr
	(ø22 mm x 10 mm)												
D. Demainsliner, Maise, Assimuses of in 94 (Desc. standard)													

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

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



Figure 3.3 (a) XRF Spectra for Plain Carbon Steel

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

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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.,

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Author Profile



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