# EVALUAREA CONFORMITĂȚII STRUCTURALE A BETONULUI PRIN COMBINAREA METODELOR VIZUALE, FIZICE ȘI CHIMICE COMBINING VISUAL, PHYSICAL AND CHEMICAL METHODS FOR EVALUATION OF RELATIVE STRUCTURAL SOUNDNESS

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Structural Health Monitoring (SHM) plays an important role in retrofitting of concrete structures. In this paper, soundness of concrete material in three domes of a structure was evaluated by the use of Schmidt Rebound Hammer test and X-ray Diffraction (XRD) test after initially investigating the whole structure visually. It has been realized that value addition in the field of NonDestructive Testing (NDT) of Civil Engineering Structures is possible only through combination of various testing techniques. By successfully combining the results of visual inspection, strength based physical test and chemical test, authors have succeeded in introducing a novel idea for further research. XRD test, which has generally not been categorized as an NDT tool for structural evaluation, gives satisfactory results when combined with a strength based NDT tool.

**Keywords:** Structural Health Monitoring; Soundness of Concrete Material; X-ray Diffraction; Visual Inspection; NonDestructive Testing; Combination of Testing Techniques.

# 1. Introduction

Concrete structure is, in its service life period, exposed to a variety of physical and chemical phenomena which may lead to its degradation. Generally, deterioration of concrete structure is marked by a fall in its strength, presence of defects such as cracks, plastic hinge, etc which result in a reduction in pH level of the material. A significant shift in the microstructure of concrete material is observed during its life period. These alterations often have detrimental effects on the service life of a structure. Constant structural monitoring thus becomes imperative so as to maintain good condition of the structure throughout its life period. In the structure is severely dilapidated, case maintenance and retrofitting techniques should be applied as per the requirement. It is well recognized that rebuilding a structure may often not be practicable. Also, it has been learnt that even design of durable structures would not be possible without complete knowledge of the mechanisms leading to its deterioration. The notion that the structural performance of a structure should be precisely predicted in order to minimize risks is becoming increasingly popular. Civil Engineers are increasingly embracing the role of "maintainers" they have not put their money into this, yet; things are starting to change [1]. Three things are vital to the prediction of structural performance of a concrete structure: 1) The realization that concrete structure

is built for a service life and the determination of this service life; 2) Quantification of the physical degradation of the structure, in terms of its strength or integrity and marking the weaker zones; 3) Study of the physical and chemical reasons for any loss in strength or integrity.

Reinforced concrete (RC) as a construction material became popular in India, as elsewhere, in the 20<sup>th</sup> Century. Majority of the buildings built in the 20<sup>th</sup> and 21<sup>st</sup> Centuries in India are therefore RC structures. In the current paper, the authors discuss a unique case wherein RC was used to complete a half-built stone structure. The structure, Taj-ul-Masajid, is one of the largest mosques in India. The serviceability of the structure has been greatly influenced by the atmosphere which is characteristic of its urban exposure. Being a historical monument, preservation of original structure is mandatory and therefore, replacement or rebuilding of the structure is not possible. Also, the cost of rebuilding would make it practically unviable.

Before being subjected to physical examination, a structure must always be evaluated visually. In the first segment of the present work, authors therefore conducted a visual investigation and selected portions within the structure for further evaluation, the details of which have been included in this paper.

Generally, a concrete structure's most valued property is its compressive strength. A number of techniques have been developed which could give

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the compressive strength of concrete. Developed in Germany in 1930, the Rebound Hammer Test is one such technique. In 1948, Ernest Schmidt, a Swiss Engineer, developed the Schmidt Rebound Hammer [2, 3]. In 1992, the Bureau of Indian Standards listed it as a standard method of NonDestructiveTesting, stating it to be a method which could be used for: 1) assessing the likely compressive strength of concrete with the help of suitable correlations between rebound index and compressive strength; 2) assessing the uniformity of concrete; 3) assessing the quality of concrete in relation to standard requirements and 4) assessing the quality of one element of concrete in relation to another. On the other hand, it stated that the estimation of the concrete strength by rebound hammer method cannot be held to be very accurate and probable accuracy of prediction of concrete in a structure is ± 25% [4]. In the present study, however, as the objective is to compare the relative soundness of concrete in three portions of the same structure, Schmidt Rebound Hammer test is an ideal method as it is convenient and easy to interpret.

In the third and most important segment of this article, authors have used the X-ray Diffraction (XRD) method to diagnose the three portions of the structure which were earlier evaluated visually and physically.

X-ray diffraction is one of the earliest and most widely applied techniques for studying the structure of solids. In the process of diffraction, electromagnetic waves of a given frequency but different phases interact to produce constructive interference (bright spots on the film exposed to the light) and destructive interference (dark spots) [5]. In 1913, W.H. Bragg and his son W.L. Bragg formulated the following relation between the spacing of atomic planes in crystals and the angles of incidence at which these planes produce most intense reflections of electromagnetic radiations, such as X-rays and gamma rays:

# $n\lambda = 2dsin\Theta$

Here, the variable d is the distance between the adjacent planes of atoms in a crystal (interplanar distance), and the variable lambda ( $\lambda$ ) is the wavelength of the incident X-ray beam, n is an integer. In this relation, which is an example of X-ray interference or X-ray diffraction (XRD), if the values of  $\Theta$  and  $\lambda$  are known, one can determine the dspacings. An X-ray diffraction pattern, which is a graph between the intensity of X-rays scattered at different angles by a sample, is plotted. A phase is a specific chemistry and an atomic arrangement and each phase has a unique "fingerprint" diffraction pattern [6]. Therefore, when properly interpreted, by comparison with standard reference patterns and measurements, this becomes a powerful tool for identification of a chemical phase. In case of a

mixture sample, the diffractogram or the diffraction pattern is a simple addition of each individual phase.

XRD tests are used to discover crystalline phases, phase distribution and also to provide quantitative analysis of identified phases. As was pointed out earlier, concrete's chemical structure tends to show variations with time. As various chemical phases would have different properties, the presence of certain phases would certainly point to deterioration. Also the presence of more varied chemical phases in the same concrete sample would signal the non-uniformity of the sample's microstructure and would therefore indicate that the sample has undergone a lot of chemical sways before arriving at its present condition. This understanding was key to the present research.

# 2. Evaluating Soundness of the Structure

The structure was evaluated in three stages:

- 1. Visual Investigation
- 2. Schmidt Rebound Hammer Test
- 3. X-Ray Diffraction Test

# 2.1 Visual Investigation

Visual investigation is one of the most influential and versatile non-destructive tools and it is typically one of the first steps in the evaluation of a structure [2]. Its effectiveness depends entirely on the knowledge of the investigator. In this research, too, visual inspection was carried out to plan the complete investigation properly. A typical investigation involves the following activities:

- 1. Perform a walk-through investigation to become familiar with the structure.
- 2. Gather background documents and information on the design, construction, maintenance and operation of the structure.
- 3. Perform a detailed visual inspection.
- 4. Plan the complete investigation.
- 5. Perform any sampling or in-place tests.



Fig. 1 - Top View of Domes.

#### Table 1

Major Defects Observed in Visual Investigation							
Structural Element	Defects Identified in Visual Investigation	Defect Area as a Percentage of Total Surface Area					
		000/					
Dome A	Dampness	20%					
	Cracking	Less than 5%					
Dome B	Dampness	Less than 10%					
	Cracking	Less than 1%					
Dome C	Dampness	50%					
	Cracking	10%					
	Spalling	25%					
	Flaking	35%					
	Blistering	35%					
	Honeycombing	15%					

#### Table 2

Compressive Strength for the Domes								
Point	Dome A		Dome B		Dome C			
Located on the Structure	Compressive Strength (N/mm²)	Average Compressive Strength	Compressive Strength (N/mm²)	Average Compressive Strength	Compressive Strength (N/mm²)	Average Compressive Strength		
		(N/mm²)		(N/mm²)		(N/mm²)		
P1	24.13	22.69	24.13	30.24	19.99	18.35		
P2	20.68		27.92		24.48			
P3	24.13		26.89		17.24			
P4	15.86		31.03		24.48			
P5	24.13		31.03		10.34			
P6	27.92		27.92		13.79			
P7	29.30		29.30		10.34			
P8	25.51		32.75		24.48			
P9	20.68		20.68		15.86			
P10	12.41		35.16		17.24			
P11	12.41		33.44		19.99			
P12	25.51		15.17		12.41			
P13	12.41		19.31		19.99			
P14	17.24		29.30		17.24			
P15	17.24		36.54		22.06			
P16	31.03		15.17		19.99			
P17	24.13		37.92		24.48			
P18	22.06		37.92		15.86			
P19	27.92		37.92		17.24			
P20	19.65		31.03		22.06			
P21	24.13		35.16		19.99			
P22	27.92		36.54		13.79			
P23	29.30		29.30		24.48			
P24	20.68		36.54		13.79			
P25	31.02		37.92		17.24			
		1				1		

In the visual investigation which was conducted on the structure, while defects were observed in many portions of the structure, it was noted that the three domes (Figure 1) were relatively more damaged. The major defects observed in visual inspection are mentioned in Table 1.

### 2.2 Schmidt rebound hammer test

One of the most widespread methods i.e. Rebound Hammer test or Schmidt Rebound Hammer test was adopted to test the domes which were initially investigated visually. In this method, the plunger of the hammer is pressed against the surface of concrete. The spring controlled mass rebounds and the extent of such rebound depends upon the surface hardness of concrete and the flexibility of the structural element. The rebound



Fig. 2 - Points of Observation for Schmidt Hammer.

value is read off along a graduated scale and is designated as the rebound number or rebound index.

The authors performed the above test by means of an N-regular type Schmidt Hammer on the inner sides of the 200mm thick slabs, the positions of which have been marked in Figure 2. To conduct the test, 25 points were marked on the circumference of the domes such that each point was at an equal distance from the previous one. Concrete surface around such points were thoroughly cleaned before taking Rebound Hammer measurements. The upper surface of the plaster was removed so that the test could be performed on the hard concrete surface. Around each point of observation six readings of rebound indices were taken and the average of these values was taken (as per IS 13311 Part 2 (1992)). The dome dimensions have also been marked in Figure 2.

With the help of the graphs provided by the manufacturer, we get the compressive strength against Rebound Number (N). If the compressive strength is in Pascal/inch<sup>2</sup>, the same can be converted into N/mm<sup>2</sup> using the following equation:

Cpsi = Cmm X 145.037

Here,  $C_{mm}$  and  $C_{psi}$  are compressive strengths in N/mm<sup>2</sup> and Pascal/inch<sup>2</sup> respectively.



Fig..5 – Compressive Strength for Dome C

Different points were located on domes A, B and C. The compressive strength for each of the located points was found using Schmidt's Concrete Test Hammer. The values of compressive strength are given in Table 2. The same are depicted through graphs in Figures 3, 4 and 5.

# 2.3 X-Ray Diffraction Test

X-Ray Diffraction (XRD) test has been used to evaluate the concrete samples collected from the three domes tested physically by means of Schmidt Rebound Hammer in order to identify different chemical phases present in each concrete sample. In the past, the test has been limited to a phase identification tool. But, authors believed that such an evaluation could help in comparison of the three concrete samples by giving deep insights into the microstructure of each, which was also the prime objective of the present investigation.

The central problem in any XRD investigation is to determine the position in space of the atoms in the unit cell of a material crystal. The material can be fine powder and then the suitable method is Xray Powder Diffraction (XRPD), or single crystal, and then the right technique would be Single Crystal X-ray Diffraction (SCXRD) [7].

SCXRD is a powerful technique that is commonly used to determine the structures of new materials. However, the technique is limited by the ability to grow nearly perfect crystals that are suitable for diffraction. For routine structural characterization of materials, X-ray powder diffraction is far more common [8].

In an X-ray powder diffraction test, monochromatic X-rays of a given wavelength,  $\lambda$ , is scattered from the material over a wide range of 20 values. The diffraction pattern or the diffractogram of a crystalline material, thus produced, provides a unique diffraction pattern of each material. The powder diffractogram consists of a series of diffraction peaks each of which is characterized by its position (20), Intensity (I) and Miller Indices (hkl) of the set of crystal planes contributing to a particular peak. The presence of many peaks, henceforth, is an indication that the crystal comprises of many different chemical phases and is, therefore, highly heterogeneous. Each X-ray reflection given by the crystal can be thought as representing a certain average sinusoidal distribution of scattering matter, or electron intensity, running through the crystal, whose amplitude can be related to the measured intensity of the reflection. By combining the distributions obtained from all the reflection we can measure, it is possible to build up a more or less complete picture of the structure [9].

In any XRD test, sample collection and preparation is a delicate process [10].

The samples for measurement were prepared in solid form. For each dome, three

locations were chosen for extraction of material. Samples were prepared so as to represent the entire structure's material composition. In case of an XRD test, the test result is in the form of a diffractogram which is used to determine the chemical composition of the material. No guantitative data is obtained and no concurrent or average values are to be determined. Therefore, XRD test of one sample representing the whole structure's composition would yield good results. In the present investigation, this was achieved by extracting material from three different locations representing the maximum, the minimum and the average compressive strength values as obtained from the Rebound Hammer test. The material thus obtained from different locations of one dome (say Dome A) was thoroughly mixed and ground in a ball mill so as to form one homogeneous powder sample. The powder sample was passed through IS sieve of mesh size 75 microns. In similar manner, powder samples for the other two domes were prepared for XRD evaluation. The samples were marked separately as D-A, D-B and D-C according to the domes they were extracted from. Each sample was homogeneously mixed prior to subjecting it to X-Ray Diffractometry. The XRD analyses were performed using CuKa radiation on a Rigaku Tabletop Diffractometer. The pulverized, homogeneous sample was side-loaded into an Aluminium sample holder. During data collection, the sample remained in a fixed position and the Xray source and detector were programmed to scan over range of 2O values (0° to 180°). Continuous scans were taken and different d-spacings and relative intensities were obtained. The results were obtained in the form of Intensity in cycles per second (cps) vs. 20 values.

Figures 6, 7, and 8 represent the peaks which were observed in the three domes, Dome A, Dome B, and Dome C, respectively. The phases have been interpreted using Powder Diffraction File Search Manual [11, 12].

Looking at the results obtained for each sample, one can find that for all three samples, the peaks indicate a predominance of Quartz (SiO<sub>2</sub>) and Calcium Oxide (CaO). The presence of Aluminium Ortho Phosphate (AIPO<sub>4</sub>) indicates the use of corrosion inhibitors by the builders. In all three samples, the presence of Iron in Ferric forms represents the occurrence of Oxidation reaction and, therefore, corrosion.

Further, the number of peaks observed for Dome A indicates that the material has a comparatively heterogeneous microstructure. The presence of sulfate based mineral points at the possibility of deterioration in the structure. Waterborne sulfates react with hydration products of tricalcium aluminate ( $C_3A$ ) phase of Portland Cement and with Calcium Hydroxide (Ca(OH)<sub>2</sub>) to form an expansive crystalline product called



Fig. 6- XRD Analysis of Concrete Material from Dome A.



Fig. 7- XRD Analysis of Concrete Material from Dome B.



Fig. 8 - XRD Analysis of Concrete Material from Dome C.

ettringite. Expansion due to ettringite causes tensile stresses in concrete. When these stresses become greater than the concrete's tensile capacity, the concrete begins to crack.

On the other hand, the sample D-B is very symmetric and fewer peaks indicate its good homogeneous condition. The homogeneity of the mixture when coupled with occurrence of fewer complex phases clearly indicates better quality of material in Dome B.

In sample D-C, the presence of many peaks and thereby, too many complex phases such as Calcicopiapite  $(Ca_2Fe_4(SO_4)_6(OH)_2.19H_2O)$  and Pigeonite ([Ca<sub>0.04</sub>Mg<sub>0.45</sub>Fe<sub>0.48</sub>]SiO<sub>3</sub>) clearly indicate a complex, asymmetric substance. Calcicopiapite, which belongs to the copiapite family, is an oxidation product of pyrite. It shows a linear topological structure [13]. The presence of copiapite and related minerals in the material is an evidence of environment impact [14]. The dissolution of calcicopiapite increases aciditiy because of ferric ion hydrolysis. Oxidation of pyrite itself is a chemical reaction that results in crystal growth of various sulfates, expansion and volume change at or near the site of pyrite. Pyrite oxidation is initiated by the presence of water and oxygen producing Fe<sup>2+</sup>. The Fe<sup>2+</sup> may be oxidized by the presence of oxidizing bacteria to form Fe3+ or a precipitate as in Fe<sup>2+</sup>SO<sub>4</sub>.nH<sub>2</sub>O or a combined Fe<sup>2+</sup>Fe<sup>2+</sup> mineral phase. The reaction involved is [15]:

2FeS<sub>2</sub> + 3H<sub>2</sub>O + CaCO<sub>3</sub> + 6O<sub>2</sub> → (Pyrite) (Water) (Lime) →Fe Ca (SO<sub>4</sub>) + 3 H<sub>2</sub> SO<sub>4</sub> + CO<sub>2</sub> + Fe (Sulfate) (Sulfuric Acid) (Iron)

## 3. Results and Discussion

In the visual inspection conducted on the entire structure, defects were identified at different locations. Among the different locations, it was observed that the domes of the structure required further detailed evaluation.

For further evaluation of the structure and to quantitatively find the variation in soundness of the individual domes, authors performed Schmidt Rebound Hammer test on the three domes. Dome C had an average compressive strength of 18.35 N/mm<sup>2</sup> which was very low when compared to Domes A and B with average strengths of 22.69 N/mm<sup>2</sup> and 30.24 N/mm<sup>2</sup> respectively. Although the compressive strength values may vary by  $\pm$  25% from the actual values, it will not affect the comparative analysis of similar structures.

For further evaluation of the material, authors used X-Ray Diffraction test. Through X-Ray Diffraction test, authors identified different chemical phases in the concrete material. This gave chemical evidence for variation in microstructural properties of material of the domes and thus proved the disparity in quality of concrete in the three domes. The test results showed that Dome A, because of the presence of sulfate based mineral phases, is susceptible to deterioration due to expansion of concrete material. Also, the test proved that it contained highly heterogeneous microstructure. On the other hand, Dome B showed highly symmetric microstructure with very few diffraction peaks. It was thus inferred that the dome is in a relatively good condition. Dome C was found to be in the poorest condition. The presence of myriad complex phases sample indicated its asymmetrical in the microstructure; the presence of complexes such as calcicopiapite highlighted the amount of chemical degradation the dome has already undergone. Copiapite, a pyrite oxidation product, greatly lowers the pH of the material. Reinforcement corrosion, which is essentially an electrochemical reaction, is facilitated by the fall in pH level of the material. The XRD test, thus, not only confirmed the results obtained in the visual inspection and physical test, but also alluded to the possible chemical reactions which may have resulted in the same.

## 4. Conclusion

Identification and quantification of structural weaknesses forms the first and most vital stage of condition assessment and retrofitting of concrete structures. While a number of techniques have been developed for the same in the past, recent research works have given much importance to the combination of different techniques. It has been found that by combining different NDT tools, one can get more comprehensive results. In this study too, three NDT methods were used to evaluate the relative soundness of concrete elements of a structure. The results of all three studies were found to be complementary. Dome B was found to be sounder than Dome A and Dome C was in the poorest condition of the three. While visual inspection pointed towards this, Schmidt's concrete test hammer gave physical proof for the same. But, the most important set of results was supplied by the XRD test. XRD test, through identification of various chemical phases in concrete, clearly indicated inferior quality of the material present in Dome C. By combining a set of NDT tools which by and large

give mutually independent results, authors have opened up new and unexplored pathways which the NDT engineers could tread in order to achieve their goals of identifying, quantifying and repairing structural weaknesses in situ. While using a combination of NDT techniques to get results and discover flaws a fortiori has been much investigated in the past, using a combination of visual, physical and chemical methods for the same is a development with great untapped potential. Further, the study reveals that X-ray diffraction, which has been used in the past mainly for phase identification

in cement samples, is a technique which could be applied practically as an NDT tool for evaluation of concrete. The technique is not a substitute for the physical tests that quantify strength. But in case of a comparative analysis of structural soundness, it can be used for identifying the possible chemical phenomena behind any significant fall in strength. However, this would require the engineers to understand the significance of different chemical phases present in a concrete. When combined with a proper strength based NDT tool, XRD test would guarantee comprehensive results.

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