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Delhi iron pillar rust characterisation by mössbauer spectroscopy analysis

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Abstract : Rust samples from the region just below the decorative bell capital of the Delhi iron pillar have been analysed by Mössbauer spectroscopy. The analysis indicates that they contain γ-FeO-OH, superparamagnetic α-FeO-OH, δ-FeO-OH and magnetite, all in the amorphous form. The Mössbauer spectrum also confirmed that the iron in the crystalline iron hydrogen phosphate hydrate (whose presence was confirmed by XRD analysis) is in the ferric state indicating that it is a stable end corrosion product.

Key words : *Delhi iron pillar rust, Mössbauer spectroscopy analysis, Rust characterisation.*

INTRODUCTION

The precise reason behind the corrosion resistance of the famous 1600 year old Delhi iron pillar (Fig. 1)[1-4] is still unknown[2-5]. In order to glean insights into the nature of corrosion products (that is responsible for its excellent resistance to atmospheric corrosion[1-5]) that forms on the Delhi iron pillar (DIP), a detailed characterization of the DIP rust was undertaken. The characterization of Delhi iron pillar (DIP) rust using X-ray diffraction (XRD)[6-7] and Fourier transform infrared spectroscopy (FTIR)[7-8] has been discussed earlier. The XRD analysis also indicated that a very small quantity of crystalline iron oxide/ oxyhydroxides was present. The results of the FTIR study confirmed the presence of phosphates, and also several different allotropic modifications of the oxyhydroxides and magnetite[8-9]. While the iron hydrogen phosphate was crystalline, it was hypothesized that the oxide/hydroxides of iron were in the amorphous form.

In order to obtain further insights in to the structural (i.e. amorphous or crystalline) and chemical aspects of the DIP rust, Mössbauer spectroscopy was utilized. Mössbauer spectroscopy is a powerful tool to understand the amorphous versus crystallinity controversy in case of the oxides/oxyhydroxides[9-11]. In case the phase that constitutes the rust is crystalline, it would lead to alignment of magnetic fields in the individual grains of the phase and provide a characteristic hyperfine splitting with characteristic magnetic fields. For example, in the case of crystalline magnetite, two sextets (10 line pattern) are to be expected in the Mossbauer spectrum at ambient temperatures. However, in case this phase is amorphous, the magnetic fields are not aligned in the individuals grains and this would result in collapse of magnetic fields or as a result of which a doublet would be obtained instead of a sextet[9-11]. A collapsed sextet would indicate the fine nanocrystalline nature of magnetite. Therefore, Mössbauer spectroscopy can be advantageously utilized to probe the crystal nature (i.e. amorphous or crystalline) of the phase. The use of Mössbauer spectroscopy for elucidation of the chemical nature of corrosion products is also well established[6, 9-11]. The aim of the present paper is to characterize the DIP rust using Mossbauer spectroscopy.