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Magnetic Property of Akaganeite Produced By Corrosion of Steels

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Abstract: *For application of cosmic-ray muon spin rotation method to inspect corrosion state of steels in large-scale architecture nondestructively, products of accelerated corrosion of steels for pre-stressed concrete method were characterized and its magnetic behavior is measured. Single phase of akaganeite, β-FeO(Cl, OH) was found predominantly in brittle lump of rust, while remaining part which keeps original form with surface rust is ferromagnetic steel inside. Magnetization of akaganeite thus produced shows broad peak between 80 and 130K with irreversibility between zero field cooling and field cooling below 270K, suggesting existence of spin frustration similar to spin glass system. Above 270K, corrosion products are paramagnetic. It means that feasibility of cosmic-ray muon spin rotation is powerful tool to investigate corrosion state of steels inside large-scale architecture.*

Keywords : *corrosion products, akaganeite, magnetization, cosmic-ray muon spin rotation, spin frustration*

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I. Introduction

Large-scale architectures for social infrastructure, e.g. highway bridges, tunnels and dams, have been exposed to severe natural environment and hardly worn for long years. Aging effects such as fatigue, damage and corrosion on steels inside concrete are worried to cause serious deterioration and accidents. In architectures constructed by the iron reinforced concrete (RC) method, a sign of corrosion on steels inside appears typically as cracks of concrete surface. For the purpose to restrain progress of corrosion, steels are covered by sheath in pre-stressed concrete (PC) method which has been widely used since the middle of 19th century. However, another problem took place: architectures made by PC method shows few precursors before serious destruction.

Cosmic-ray muon spin rotation (μSR) is promising new method for inspection of chemical and physical states of steels inside large-scale architectures [1]. Recently, it is demonstrated that in pure iron the μSR signal with ~50MHz in frequency can be distinguished by decomposition analysis based on different life time of positive and negative muons consisting of about 70% of cosmic-ray. Parameters of rotation frequency and relaxation rate were confirmed by using intensive muon source in Paul Scherrer Institut (PSI) and the similar μSR signals were observed in various steel bars (SB) which are commercially available for practical use, but with much faster relaxation characteristic for RC (SBRC) and PC (SBPC) respectively [2]. For the purpose to clarify feasibility to detect progress of corrosion based on change of magnetism, μSR studies on accelerated corrosion are in progress. In this work, we report characterization and magnetism of materials produced by corrosion of SBPC.

II. Sample Preparation and Characterization

Electrochemical accelerated corrosion in chloride solution was performed on commercially available SBPC by Neturen Co. Ltd. Details of the method and sample composition are described elsewhere [3]. Sample provided after corrosion of 8 days are shown in Figure 2.1. Roughly speaking, sample is categorized into three parts: part a) steel bars which keep original form with surface rust, part b) brittle lumps of rust which were found in gaps of twisted steel bars and part c) frothy precipitates melt in chloride solution.

Figure 2.1 PC steel bar after accelerated corrosion for 8 days.

Figure 2.2 shows powder XRD pattern of part b, brittle lumps of rust, by using Cu K_α source. It precisely coincides with typical pattern of akaganeite, β-FeO(Cl, OH). It is emphasized that there are no other peaks unassigned to akaganeite. Akaganeite is known to be formed on the part of thick rust layers on carbon steels exposed to air in marine environment [4, 5, 6]. Single phase of akaganeite was produced when the initial concentrations [Fe^{2+}] and [Cl^-] are high and stoichiometric ratio of 1:2 [5].

Figure 2.3 shows FTIR spectra taken by Shimadzu FTIR-8700. It shows typical pattern in β-FeOOH but apparently different from those observed in α-Fe_2O_3 and γ-Fe_2O_3 nanorods [7]: peaks at around 650 cm^{-1} and 700 cm^{-1} represent characteristic vibrations of Fe-O, and weak peak at 850 cm^{-1} was attributed to OH bonding in β-FeOOH. From XRD and FTIR spectra, the brittle lumps of rust which were produced by electrochemical corrosion in chloride solution are predominantly assigned to single phase of akaganeite predominantly.

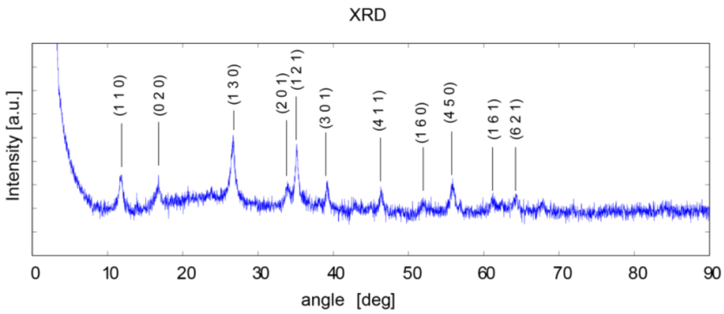

Figure 2.2 Powder XRD patterns of brittle lumps of rust after 8 days accelerated corrosion of PC steel.

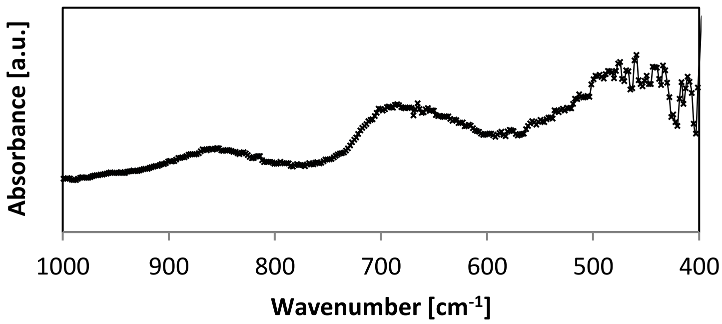

Figure 2.3 FTIR spectra of brittle lumps of rust after 8 days accelerated corrosion of PC steel.

In XRD pattern of part c, frothy precipitates melt in chloride solution, there was no peak which exceeds background noises.. Contribution from part c is considered negligible in μSR measurement. On the other hand, in part a, steel bars which keep original form, typical μSR signal at around 50 MHz was observed, indicating that inside of steel bars covered by thick rust is ferromagnetic. Details will be discussed elsewhere [8]. Here we concentrate on magnetism of akaganeite.

III. Magnetization of Akaganeite

Magnetizations in zero field cooling (zfc) and field cooling (fc) between 4K and 300K are measured by using SQUID magnetometer in CROSS laboratory in Neutron Science and Technology Center. Low magnetic field magnetization under 1 Oe and 5 Oe are shown in Fig. 3.1. There seems constant magnetization independent on temperatures and magnetic field. Although a finite composition of steels or other components were not found in XRD nor FTIR, a possibility of contamination of samples by ferromagnetic component is not neglected in magnetization measurement in low magnetic field. We consider it reasonable to remove ferromagnetic contamination to see magnetization of akaganeite from total magnetization so that magnetic susceptibility at 300K coincides with each other as shown in Fig. 3.2. It shows broad peak between 80 and 130K in zfc with increasing temperature. FC susceptibilities increase monotonically against decreasing temperature. At 5 Oe, kink is observed above 80K. Overall aspects suggest existence of spin fluctuation typically observed in spin glass. μSR measurements below 80 K support existence of spin-glass-like ordering whose relaxation functionis best fitted by $A\exp(-\lambda t^{-0.5})$, where A is amplitude and λ is relaxation rate [8].

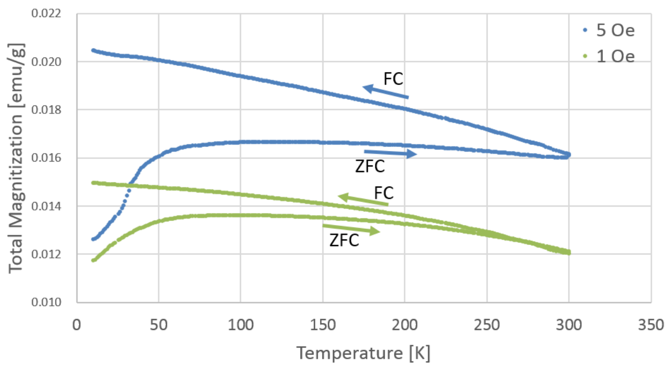

Figure 3.1 Total magnetization of corrosion product by electrochemical corrosion of steel in chloride solution.

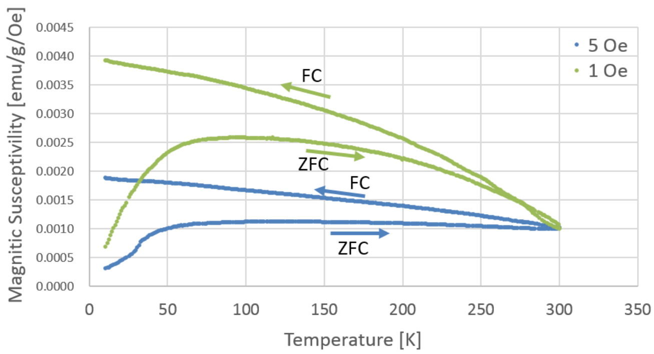

Figure 3.2 Magnetic susceptibility of akaganeite, b-FeO(Cl, OH) produced by electrochemical corrosion of steel in chloride solution.

Comparing with magnetization curves observed in ultrafine akaganeite nanoparticles in high magnetic field, peak temperature where zfc magnetization shows maximum, and irreversible temperature where irreversible separation between zfc and fc takes place, are much higher than our results, while overall aspects are similar [9]. This indicates that magnetic transition temperature depends on size as well as impurity element.

IV. Conclusion

We characterize corrosion products of PC steel by electrochemical corrosion in chloride solution. By XRD and FTIR measurements, predominant corrosion products in brittle lumps of rust is assigned as single phase of akaganeite, FeO(Cl, OH). Magnetization under low magnetic field suggests existence of spin fluctuation typically observed in spin glass below 80K. These results strongly support our expectation to investigate corrosion state of steels in large-scale architecture nondestructively by using cosmic-ray muon spin rotation method.

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