Electromagnetic Properties of Iron Oxide Corrosion Product Powders at Radio Frequencies

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***Abstract* — We calculated the refractive index from S-parameter data for corrosion samples tested in waveguide from 12.4 GHz to 110 GHz. This approach minimizes the modal noise often seen in derived permittivity and permeability for low-loss samples in this range. Also, any resonance absorption due to atomic structure is made clearly discernible. We are interested in observing spectral signatures with network analyzer measurements to quantify corrosion in reinforcing steel embedded in concrete. Here, we report room-temperature measurements of the refractive index for magnetite, maghemite, goethite, hematite, calcium ferroaluminate, and bridge girder corrosion powders, all of which exhibit flat spectral responses.**

***Index Terms* — Corrosion, iron oxide, powder, radio frequencies, refractive index.**

I. INTRODUCTION

At the National Institute of Standards and Technology (NIST), we are characterizing material parameters of corrosion for spectral shapes that could be used to nondestructively quantify the extent of corrosion on reinforcing steel bar (rebar) in concrete bridge decks. Our effort is part of a larger project that is also exploring the antiferromagnetic resonance (AFMR) associated with hematite and goethite iron oxides. AFMR results obtained to date are at temperatures well below frozen water and at millimeter wave frequencies [1]. We seek alternative spectral signatures useable at lower frequencies and within ordinary seasonal temperatures that will be more useful for developing systems for quantification of early rebar corrosion. Work of other authors suggests that this early stage corrosion on steel embedded in concrete is largely magnetite [2].

II. THEORY

Previously, we have derived permittivity and permeability data for many powders related to corrosion using the transmission/reflection method [3], and found these to contain many measurement artifacts due to geometric resonances and higher-order modes. These noise effects are accentuated in low-loss samples and several of our samples have low loss. To deal with this issue we have found that the index of refraction provides a more reliable derived parameter.

Low-loss materials exhibit standing-wave resonances in the measured reflection and transmission coefficients at frequencies where $L = m\lambda/2$ (m is an integer and λ is the wavelength). This geometric resonance complicates finding any intrinsic resonances in the reflection and transmission data, but also allows the possibility of excited higher-order modes from imperfections and sample inhomogeneities. When electromagnetic waves are used for nondestructive applications, the derived parameters must be robust and the refractive index is clearly desirable.

When geometric resonances occur in the extracted permittivity ε, permeability μ, and wave impedance ζ, the refractive index n extracted from the same reflection and transmission data proves to be smoother and easier to interpret [4]. Our data support this and also show that the effects of standing waves are reduced.

The complex relative permittivity ε_r and permeability μ_r are given by

$$\varepsilon_r = \varepsilon_r' - j\varepsilon_r'' \tag{1}$$

$$\mu_r = \mu_r' - j\mu_r'' . \tag{2}$$

We assume in this paper that the iron oxide and corrosion samples under examination have losses low enough so that $\varepsilon'_r, \mu'_r >> \varepsilon''_r, \mu''_r$. Therefore,

$$n = n' - jn'' \cong \sqrt{\varepsilon_r' \mu_r'} \left(1 - \frac{j}{2} \left(\frac{\varepsilon_r''}{\varepsilon_r'} + \frac{\mu_r''}{\mu_r'} \right) \right). \tag{3}$$

Note that the dispersion characteristics of ε'_r, and μ'_r appear primarily in the real part of the refractive index, and the imaginary part depends primarily on the dielectric and magnetic loss factors ε''_r and μ''_r and therefore on the loss.

III. TEST SAMPLES

We tested magnetite ($FeO.Fe_2O_3$), hematite (α-Fe_2O_3), and goethite (α-FeOOH) pigment powders, and synthetic maghemite (γ-Fe_2O_3) powder. These powders all have purities of greater than 90% and particle sizes of 1 µm to 5 µm.

To acquire the reflection and transmission coefficients, we used the following waveguide bands: WR62, WR42, WR15 and WR10. The powders were mixed in a low-loss matrix material that was either beeswax, for the WR62 fixture, or perfluoropolyether (PFPE) oil, for all of the other fixtures. The refractive indices we derive for beeswax and PFPE oil are in the range of 1.5 and 1.42, and so do not dominate the behavior of the extracted refractive indices of the iron oxide and corrosion powders under test. Powder loading ranged from 17 % to 25 % by volume. Results in Fig. 1 are normalized to 20 % loading.

In addition, dense flakes of corrosion were collected from a bridge girder that supported the concrete deck of a bridge removed along US highway 34 in Rocky Mountain National Park, CO. Based on visual inspection, these came from points of contact between the deck and girder, positions which imitate the low-oxygen, low-water environment of rebar corrosion before cover cracking. The flakes were ground and sieved to < 38 µm dimensions. We also measured calcium ferroaluminate (C_4AF) powder, a ferrimagnetic compound in cement.

IV. MEASUREMENT RESULTS

In Figs. 1(a) and (b), we plot *n'* and *n"* of the powders, computed with the algorithm in [3]. These match up relatively smoothly across the waveguide bands, and slight discontinuities across the waveguide bands are within systematic error range. For the measured data, Type B expanded uncertainty for *n'* is U = k uc = 0.2 (k = 1) at 60 GHz, where k is coverage factor. Magnetite has the largest *n'* and is the most lossy iron-oxide powder component. Other iron-oxide powders have relatively lower loss, with girder corrosion data being intermediate. We are planning to complete the measurement from 33 GHz to 50 GHz by the time of the conference.

V. CONCLUSION

As shown in [2], we found that magnetite has a spectral shape between 12.4 GHz and 26.5 GHz that may be useable for corrosion quantification. It exhibits the highest loss and this loss remains constant out to 110 GHz, which may provide contrast in broadband imaging. Our bridge girder results continue to support magnetite as a major constituent in early rebar corrosion.

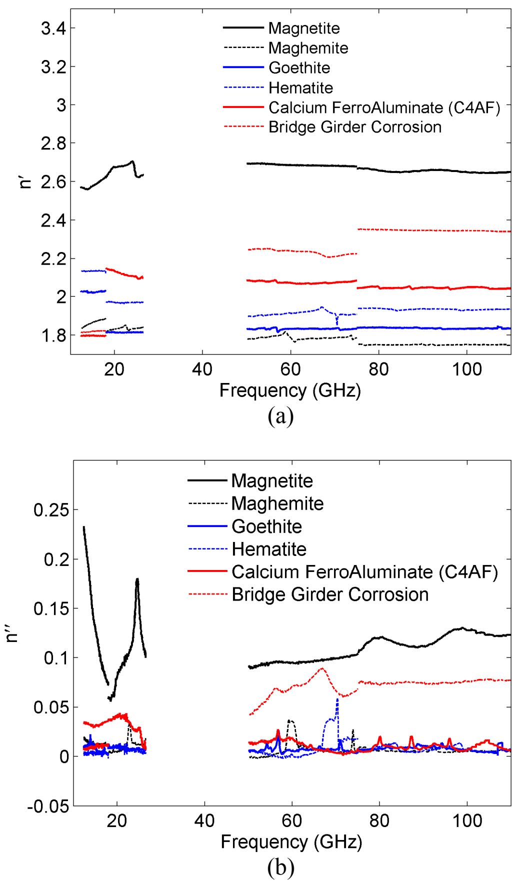

Fig. 1. Refractive indices of the iron oxide powders, C_4AF and bridge girder corrosion powder: (a) real and (b) imaginary parts.

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