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RAMAN SPECTRA OF POSSIBLE CORROSION PRODUCTS OF IRON.(U)
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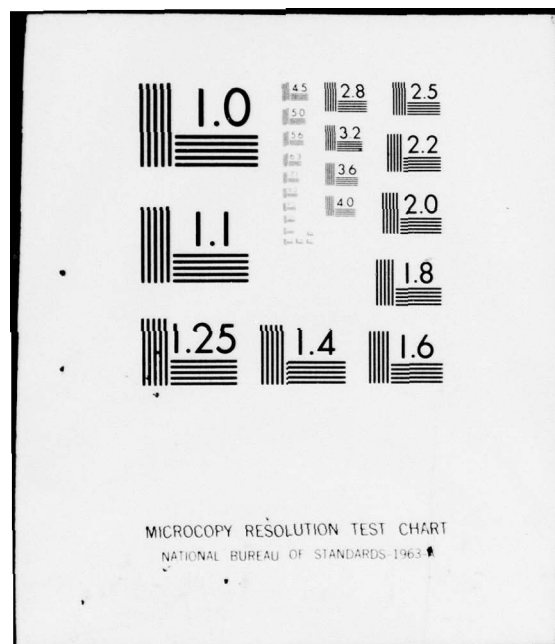
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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Raman spectra of FeO , Fe_3O_4 , $\alpha\text{-Fe}_2\text{O}_3$, $\alpha\text{-FeOOH}$, and $\gamma\text{-FeOOH}$, the common products of iron oxidation, have been measured. The spectra of FeO and Fe_3O_4 appear to be identical. Armco iron oxidized in air at 250°C was examined by Raman spectroscopy after varying periods of exposure and the surface film was found to contain Fe_3O_4 and $\alpha\text{-Fe}_2\text{O}_3$. α This manuscript has been submitted for publication in Applied Spectroscopy.		

INTRODUCTION

Laser Raman spectroscopy can be used for in situ analysis of corrosion product films on metal surfaces in gaseous and aqueous environments (1). The technique can provide quick and unambiguous identification of compounds present in surface films as thin as 50 Å (2). Since water and most gases give very weak Raman spectra, there is little spectral interference by the corrosive environment, and metal samples can be examined undisturbed while corrosion proceeds.

In order to identify compounds resulting from metal oxidation it is, of course, necessary to have spectra of the pure compounds available for reference. There can be considerable variation in the intensity of Raman scattering from one compound to another. For in situ Raman spectroscopy to be a useful technique in the study of thin corrosion films, the corrosion products must have reasonably strong spectra. Very few spectra of structural metal oxides have been published, so the first step in such a study must be to record spectra of the compounds of interest.

Iron is by far the most important metal for corrosion research. The thin oxide films it forms in most environments allow its use under nearly all conditions imaginable; however, the composition of such films is not well understood. There is experimental evidence evidence that the products of iron oxidation in air and in pure oxygen are FeO, Fe₃O₄, α-Fe₂O₃, γ-Fe₂O₃, α-FeOOH, and γ-FeOOH (3-7). The products of aqueous corrosion have been reported as Fe₃O₄, γ-Fe₂O₃, α-FeOOH, and γ-FeOOH (8-11). To use Raman spectroscopy to analyze surface films on iron, spectra of these compounds are required but only that of α-Fe₂O₃ is available in the literature (12). In this study, we have recorded Raman spectra of polycrystalline samples of the above named iron compounds and used them to identify corrosion product films to demonstrate the capability of this experimental technique in characterizing oxide films on iron surfaces.

EXPERIMENTAL

Several oxides of iron are readily available as reagent grade powders. Ferrous oxide, FeO , was purchased from Pfaltz and Bauer, Inc., Fe_3O_4 , from Amend Drug and Chemical Company, and $\alpha\text{-Fe}_2\text{O}_3$, from Alfa Products, Inc.

Crystalline $\alpha\text{-FeOOH}$ was precipitated by hydrolysis of 0.1 M ferric oxalate solution at the initial pH of 6.5 (adjusted by the addition of 1 M NaHCO_3 solution) (13,14). The solution was held at 100°C for 45 minutes and allowed to cool. The resulting precipitate was washed with distilled water, separated from the solution by centrifugation, and dried at room temperature.

Hydrolysis of a ferrous chloride solution formed $\gamma\text{-FeOOH}$. A solution of 20 g $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ in 500 ml of water was added to 100 ml of 2 M hexamethylenetetramine solution, giving a blue-green $\text{Fe}(\text{OH})_2$ precipitate. To this was added 100 ml of 1 M NaNO_2 solution and the mixture was held at 60°C for three hours (15,16). After cooling, the rust-colored precipitate was removed by filtration, washed with water, and dried at 100°C . The identity and purity of the compounds synthesized were confirmed by their infrared absorption spectra (17).

Raman spectra were recorded with a Spex Industries Model 1401 double monochromator using a photon counting detection system. A Coherent Radiation Laboratories Model 52 argon ion laser was used as the excitation source. Both the 488.0 and 514.5 nm wavelength laser lines were used and the power at the sample was approximately 500 mW. In the sample compartment of the spectrometer, the ion compounds, in the form of KBr-iron oxide pellets, were held at a 20° angle from the vertical laser beam. The angle of incidence of the beam has a strong effect on the intensity of the Raman scattered light collected from a flat, opaque surface, so the angle was chosen to give the most intense spectra possible (2). To examine surface films on Armco iron samples, the same arrangement was used. Flat, rectangular iron samples were held at the same 20° angle from the incident beam.

Armco iron samples were mechanically polished and washed with distilled water before being placed in a preheated laboratory furnace. After high temperature exposure for a specified time, the samples were allowed to cool to room temperature and Raman spectra of their surfaces were recorded.

RESULTS AND DISCUSSION

In addition to identifying specific compounds, vibrational Raman spectra can be used to identify different crystalline forms of the same compound. This is a valuable asset in the study of iron oxidation because nearly all the oxides of iron can exist as two or more polymorphs. The spectra of the two most common forms of FeOOH are shown in Figure 1. Although their chemical compositions are the same, different crystal structures and, therefore, different bonding and symmetry cause very different Raman spectra. The strong bands at 397 ($\alpha\text{-FeOOH}$) or 252 cm^{-1} ($\gamma\text{-FeOOH}$) immediately identify the two oxyhydroxides; no other iron oxide has been found to have bands at these positions.

The Raman bands observed from iron oxides are listed in Table I. As shown in Figure 2, the spectra recorded from FeO and Fe_3O_4 samples are identical. The samples consist of two distinct compounds, as confirmed by their X-ray diffraction patterns, but the spectra are the same. It may be that the vibrations which cause Raman bands in Fe_3O_4 are due to Fe-O bonds which are nearly identical to those of FeO , but we believe a more likely explanation is that the FeO , which is unstable at temperatures below 570°C (18), is decomposed by the laser. The problem of sample decomposition is a common one when opaque samples are examined using relatively intense laser excitation. Visible light is strongly absorbed by black FeO , and heating due to the absorption of the excitation beam may transform it into Fe_3O_4 and Fe , the stable species at room temperature. Both Fe_3O_4 and FeO are black and present problems with decomposition in the laser beam. After a period of exposure, spectral intensity decreases and the surface of a pellet of either compound may be visibly changed. The

Decomposition due to the excitation beam seems to be less of a problem with the red-brown compounds, α - and γ -FeOOH and α -Fe₂O₃. Probably because decomposition is slower and absorption is less, the red-brown compounds give more intense spectra than Fe₃O₄. The difference in spectral intensity is demonstrated in Figure 3, which shows the spectrum of a mixture of 90% Fe₃O₄ and 10% α -Fe₂O₃ by weight. In spite of the concentration difference, the bands due to ferric oxide, α -Fe₂O₃, are as strong as those of Fe₃O₄. Because of the wide variation in spectral intensities among these compounds, it is not possible to determine exactly a single minimum concentration necessary to observe the spectrum of a minor component in a mixture. As little as 5% α -Fe₂O₃ can be identified in Fe₃O₄ but, in a mixture with Fe₃O₄ as the minor component, at least 30% Fe₃O₄ is required before it can be seen in α -Fe₂O₃. The intensities of the FeOOH spectra are midway between Fe₃O₄ and α -Fe₃O₄ so their minimum detectable concentrations are between 5 and 30%.

Due to the simplicity and relatively large spacing of bands in the Raman spectra of iron oxides, identification of compounds in a mixture is not difficult. Unlike the broad, overlapping bands of infrared spectra of these compounds (19), the relatively narrow Raman bands allow immediate identification of compounds in a mixture.

As an example of the ability of Raman spectroscopy to characterize corrosion product films on iron surfaces, we have examined polished Armco iron plates which were oxidized in air in a laboratory oven. Spectra resulting from oxidation at 250°C are shown in Figure 4. The Raman spectra identify the surface film as Fe₃O₄ and α -Fe₂O₃. The Fe₃O₄ bands at 616 and 663 cm⁻¹ can be seen in all three spectra, while at first only the strongest Fe₂O₃ bands can be seen. After 217 hours of exposure all seven bands of α -Fe₂O₃ are visible. Bands due to both compounds increase in intensity throughout the exposures, indicating that both the Fe₃O₄ and α -Fe₂O₃ layers are thickening.

Raman spectra of surface films as thin as 50 Å on a metal surface have been reported (2), but the minimum film thickness necessary for compound identification depends on the Raman scattering strength of the film. After oxidation under conditions similar to those reported here, Poling (19) found an oxide film thickness of approximately 500 Å for a 50 hour exposure at 250°C. An α -Fe₂O₃ film of 100-200 Å is probably necessary as a minimum before spectra useful for identification can be obtained with a single scan; for a thinner film the bands are not discernible above the noise. For other compounds, the minimum film thickness can vary greatly, depending on Raman scattering characteristics.

CONCLUSIONS

Raman spectra of the oxides of iron can be obtained with little difficulty using conventional instrumentation. The spectra can be used to identify the compounds and to specify which polymorphs of the compounds are present in thin films on iron surfaces. Spectra of thin surface films are identical to those of the pure compounds comprising the films.

There is considerable variation in intensity of Raman scattering from these oxides. The black compounds, FeO and Fe₃O₄, give weak spectra, whereas those that are lighter in color, α -Fe₂O₃, α -FeOOH, and γ -FeOOH, give stronger spectra. Decomposition of samples in the laser beam is a problem with FeO and Fe₃O₄ and, possibly, the use of techniques to minimize decomposition such as rotating the sample or using different wavelength excitation might give stronger spectra. These two oxides have been found to give identical spectra. While the reason for this is not known, it is proposed that the FeO is decomposing in the laser beam to give the more stable Fe₃O₄.

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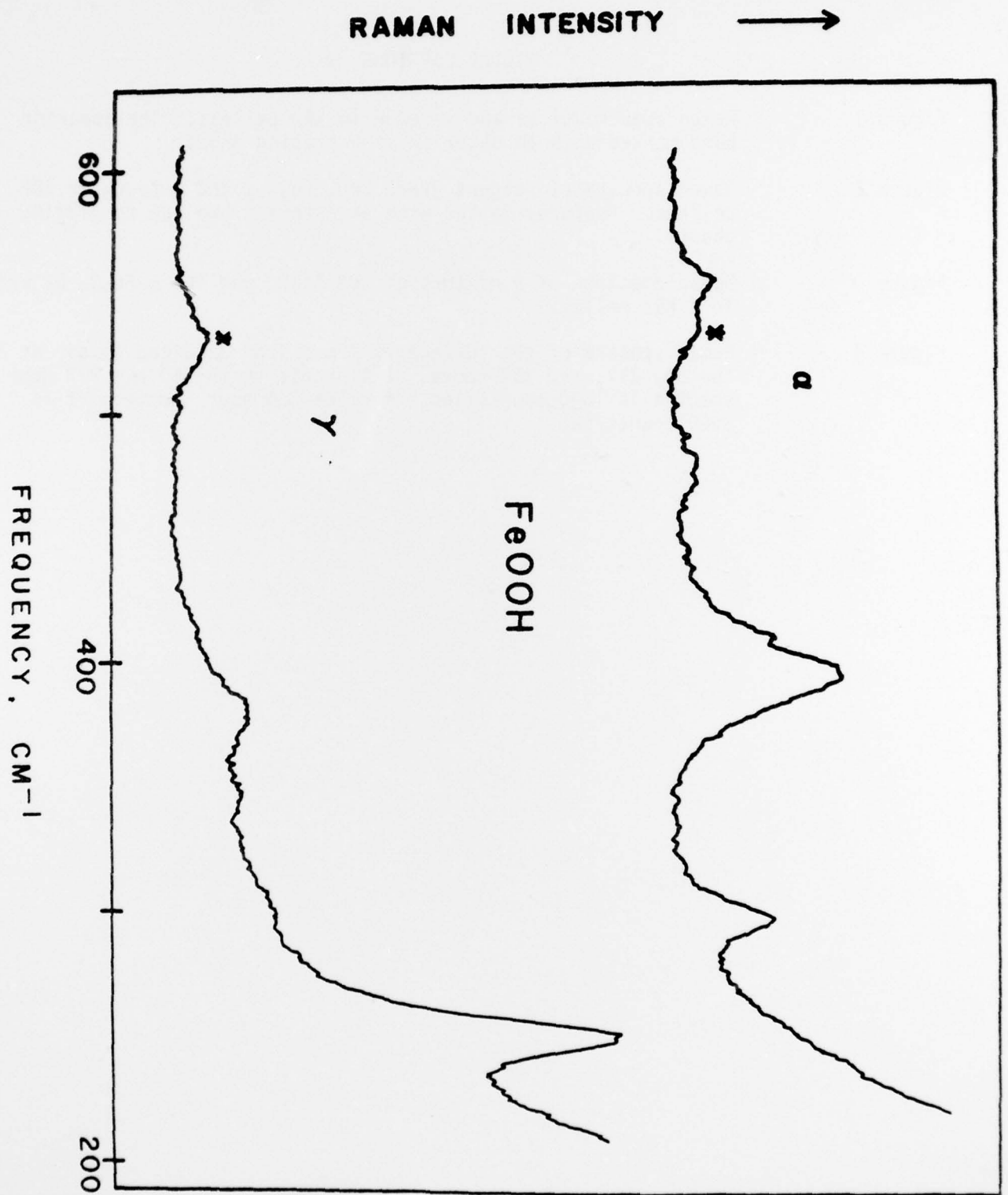
TABLE I

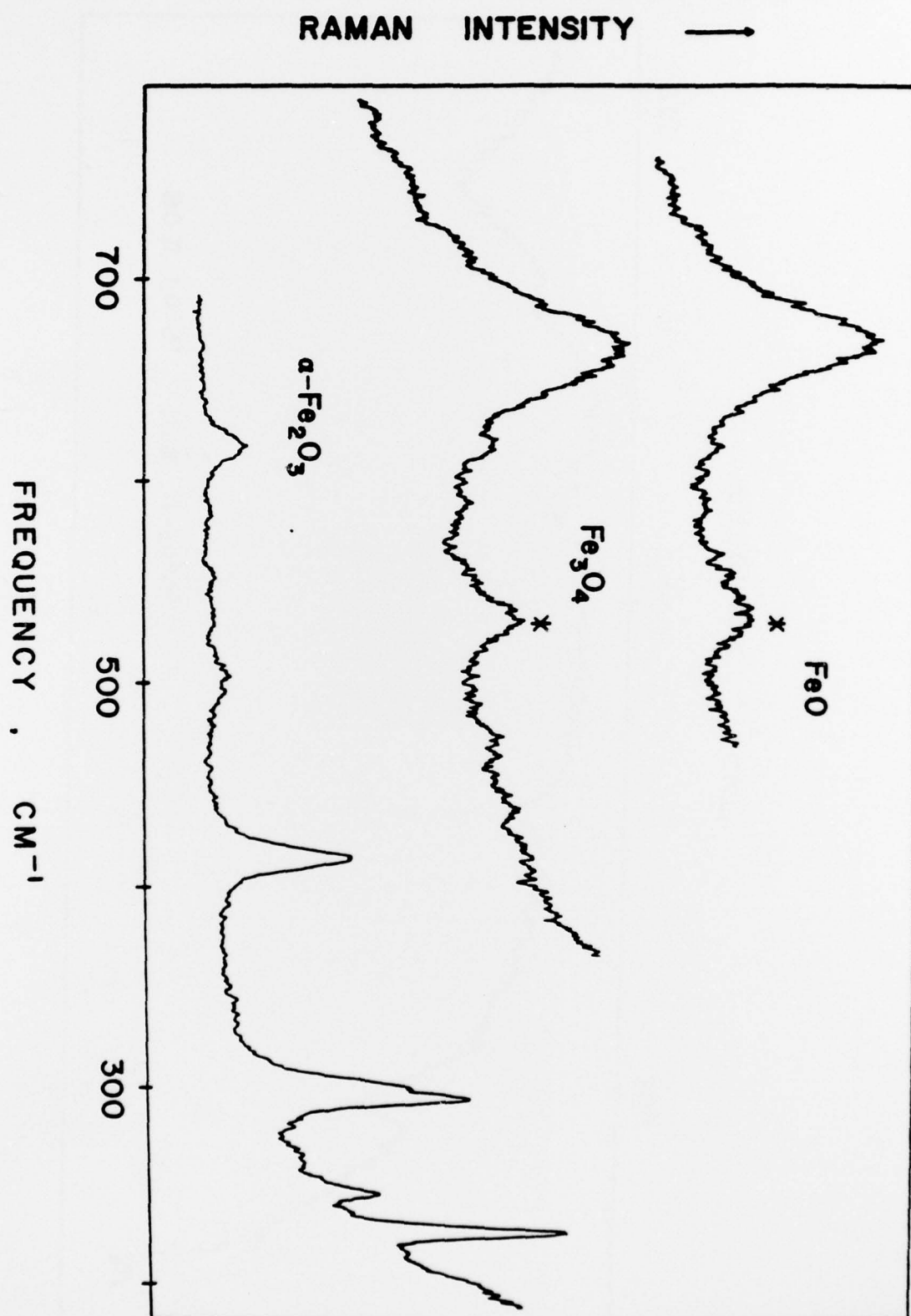
Frequencies Of Iron Oxide Raman Bands

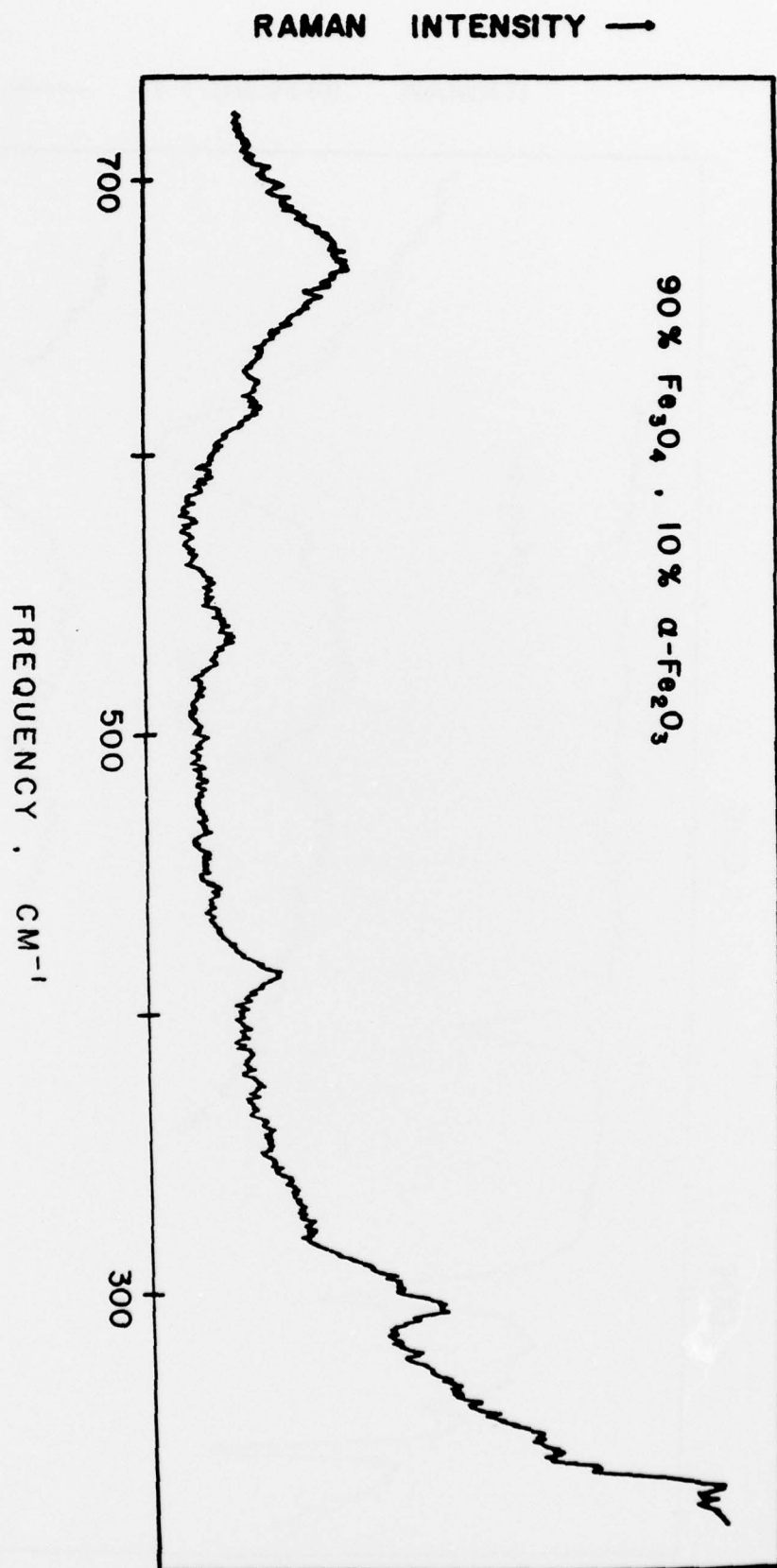
<u>Compound</u>		<u>Band Positions (cm⁻¹)</u>				
α -FeOOH	goethite	298	397	414	474	550
γ -FeOOH	lepidocrocite	252	380			
FeO	wüstite	616	663			
Fe ₃ O ₄	magnetite	616	663			
α -Fe ₂ O ₃	hematite	227	245	293	298	414
		501	612			

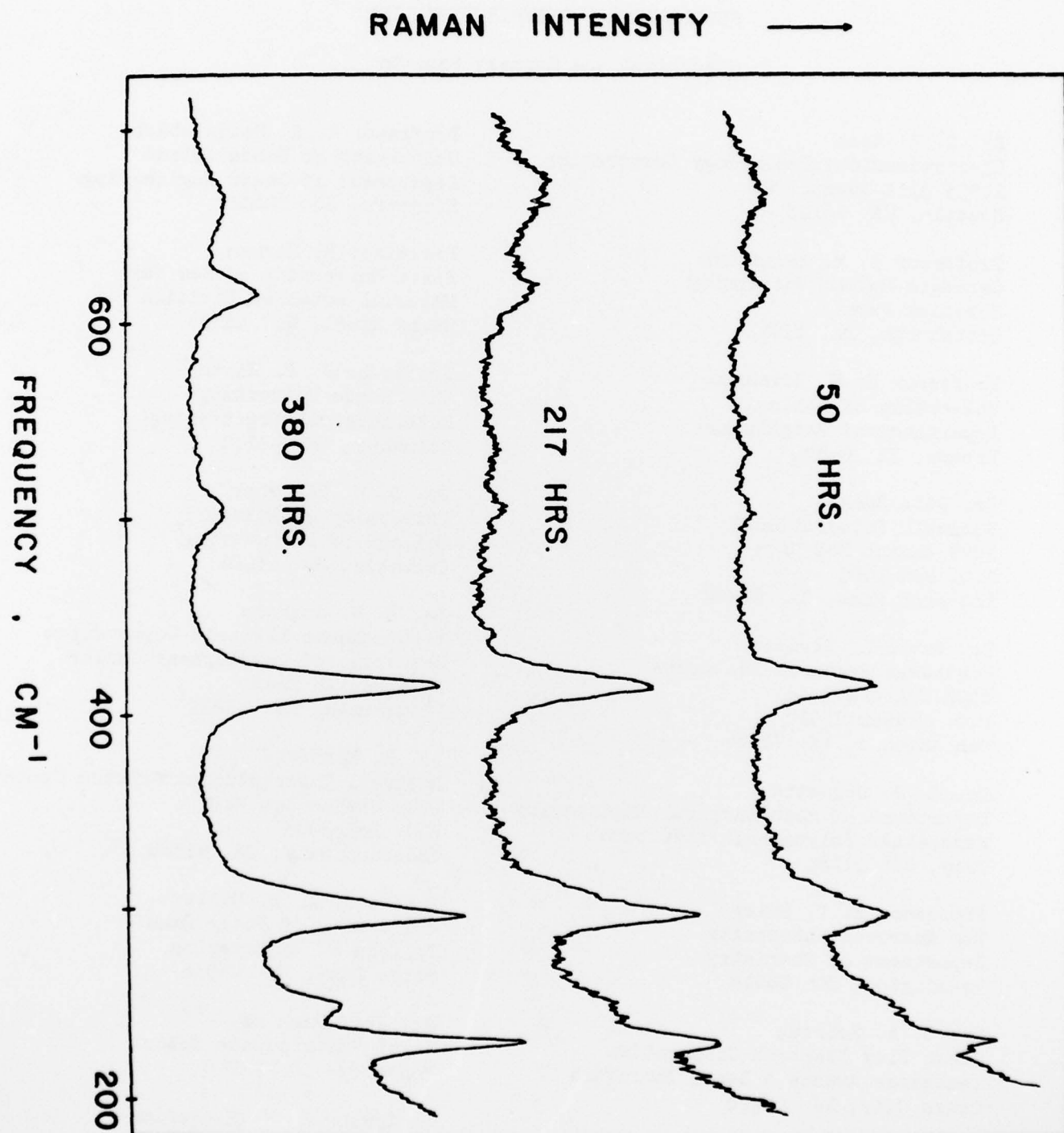
FIGURE CAPTIONS

- Figure 1 Raman spectra of α - and γ -FeOOH in KBr pellets. The apparent band marked with an asterisk is a grating ghost.
- Figure 2 Raman spectra of reagent grade FeO, Fe₃O₄, and α -Fe₂O₃ in KBr pellets. Features marked with an asterisk are due to grating ghosts.
- Figure 3 Raman spectrum of a mixture of 90% Fe₃O₄ and 10% α -Fe₂O₃ by weight in a KBr pellet.
- Figure 4 Raman spectra of the surface of Armco iron oxidized in air at 250°C for 50, 217, and 380 hours. Full scale on the 50 and 217 hour spectra is 1000 counts/sec., for the 380 hour spectrum it is 3000 counts/sec.









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