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Application of Impedance Spectroscopy Models to Measurement of Oxide Thickness of Stainless Steel

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The suspension assembly component of a disk drive consist of a precision stainless steel spring (303 free machining) with integrated high bandwidth copper interconnects, Figure 1. The manufacturing process for this component exposes the steel to environments that alter the oxide layer significantly. Variations in the oxide can give rise to layer-to-layer adhesion problems and corrosion issues.

Figure 1 Stainless steel suspension assembly with integrated high bandwidth copper interconnects used in a hard disk drive.

Characterization of the oxides has been done using XPS and impedance spectroscopy. While impedance spectroscopy is very sensitive and can provide very quick turnaround times, interpretation of the results in the context of needed process capabilities can be problematic. XPS can provide more detailed chemical information but is much more expensive and requires significant time.

The experimental procedure to obtain impedance spectra has been designed to minimize surface current distributions due to ohmic effects. A boric acid buffer solution with conductivity of 11.2 mS/cm and a pH of 7.2 was used. This is about 22 g/L boric acid and sufficient NaOH to adjust the pH to the desired value. Boric acid is used because it is relatively non-interacting with the stainless steel oxide to be measured. The sample area exposed to the electrolyte was circular and 0.071 cm^2, as small as practical to minimize effects of current distribution. It was produced by tape mask punched to expose the steel of interest. The slight recess of the mask may also provide additional benefits by creating a recessed electrode.

Graphical interpretation of impedance spectra[1] indicate that the data is readily interpreted by a constant phase element, Figure 2, for the range of frequncies sampled. Using the value of Q from the CPE parameters to estimate oxide thickness assuming capacitive interpretation of Q results in an unsatifactory value (Table 1).

XPS analysis of the oxide indicates there are significant differences between the samples, Figure 3-4, Table 1. The composition changes through the oxide thickness where the first sample is rich in Cr and the second is rich in Fe and Ni. There is advantitious carbon on the surface of both samples.

The compositional changes indicate that the CPE behavior could be interpreted as a normal distribution of impedance as opposed to a surface distribution. Hirschorn et al[2] have recently provided a film thickness interpretation of impedance data exhibiting CPE behavior. They have used a power law model of varying resistivity for a normal distribution of impedance that was calibrated to XPS data for a 17Cr stainless steel. Using the parameters of Hirschorn to interpret the CPE data obtained for this steel, a remarkably accurate estimate of oxide thickness is obtained, Table 1.

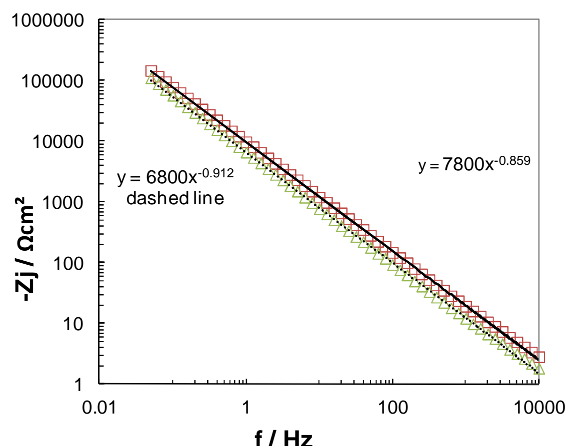

Figure 2 Imaginary part of the impedance for two stainless steel samples.

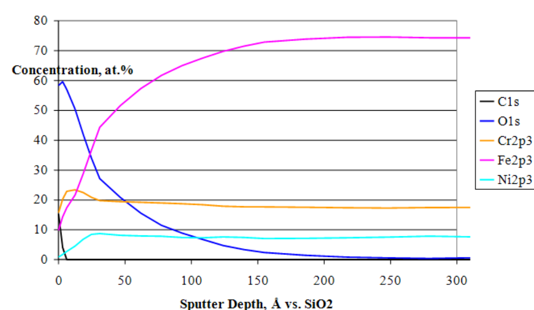

Figure 3 XPS depth profile of oxide corresponding to impedance spectra with alpha equal to 0.912.

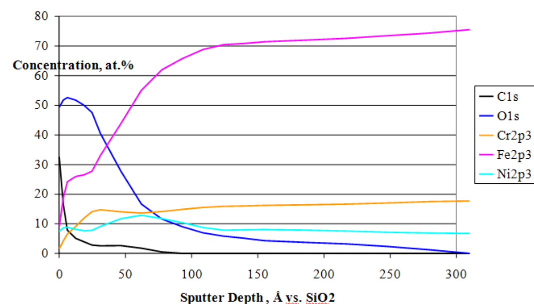

Figure 4 XPS depth profile of oxide corresponding to impedance spectra with alpha equal to 0.859.

Table 1 Oxide thickness of stainless steel samples by various methods.

	Ceff = Q	Hirshorn	XPS
Sample 1 α=0.912	0.38 nm	2.4 nm	2.8 nm
Sample 2 α=0.859	0.54 nm	4.8 nm	4.9 nm

Interpretation of impedance data of 303 free machining stainless steel exhibiting CPE behavior using the Hirschorn model has been useful to measure the effectiveness and repeatability of the treatment of oxide films on stainless steel through the various processes used in the manufacture of suspension assemblies.

References

1. M. E. Orazem and B. Tribollet, *Electrochemical Impedance Spectroscopy.* Hoboken, NJ: John Wiley & Sons, 2008.
2. B. Hirschorn, M. E. Orazem, B. Tribollet, V. Vivier, I. Frateur, and M. Musiani. *J. Electrochem. Soc.* 157 (2010): C452-C457.