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MAGNETIC PROPERTIES OF A NOVEL IRON CARBIDE FILM,
Fe₇C₃, FORMED IN A GLOW DISCHARGE

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Plasma enhanced chemical vapor deposition offers considerable promise for producing unique metal and ceramic structures. Notable features of this deposition technique include low temperature processing, ease of reproducibility, the preparation of unusual phases, the production of either amorphous or oriented crystalline films, and the alteration of film properties by using different process conditions and post production thermal treatment. The iron-carbon-oxygen system has been used to deposit a number of unique films, some of which may have useful magnetic properties. This paper describes magnetic and Mössbauer effect studies of plasma deposited Fe₇C₃ thin films.

Thin film coatings of iron carbide deposited on glass microscope cover slips were made by introducing Fe(CO)₅ with H₂ carrier gas into a plasma reactor.¹ X-ray diffraction showed the resulting films to be Fe₇C₃. Hysteresis loops and magnetization curves, obtained by using a SQUID magnetometer, gave film coercivities ranging from 280 Oe at 300K to 1130 Oe at 10K. In contrast, the coercivity of bulk Fe₇C₃ is about 35 Oe.²

Mössbauer spectra were obtained at 295 and 78K by using a Harwell constant acceleration spectrometer, a room temperature rhodium matrix cobalt-57 source, and room temperature α -iron foil for calibration. Figure 1 shows the Mössbauer spectra and fits for the thin film sample of Fe₇C₃. The solid line is a least squares fit to the data using a model having four different magnetic iron sites. In preliminary fits, all 2,5 sextet lines had very low intensity, and in the fits shown in Figure 1, all 2,5 lines were constrained to have zero intensity.

The Fe₇C₃ spectra exhibit smaller splittings, and hence lower hyperfine fields and average magnetic moments, than does α -iron. More important, the more complex spectra are indicative of a more complex crystal structure, and provide evidence for an orientation of the iron magnetic moments normal to the film plane. Four separate magnetic sextets, corresponding to four magnetically inequivalent iron sites, are required to satisfactorily fit the spectra. In addition, the 295 K spectrum shows the presence of 7% by area of a quadrupole doublet, which presumably results from a superparamagnetic component. This component is ordered at 78K, where it contributes mainly to the magnetic component with a small field of 108 kOe. These fits are consistent with the Fe₇C₃ structure. Three of the sextets correspond to the different iron sites in Fe₇C₃.³ The other iron site probably corresponds to crystalline defect layers found in

Fe₇C₃.⁴ The Fe₇C₃ film is similar to bulk Fe₇C and Fe₃C₂ in net magnetic moment, but is quite different in terms of local site coordination number and environment.

It is not possible to fit these spectra with the areas of each sextet constrained in a 3:2:1:1:2:3 ratio, as would be the case for a film in which the magnetic moments were randomly oriented with respect to the direction of the γ -rays. The observed ratio is close to 3:0:1:1:0:3, the ratio expected if the iron magnetic moments are parallel to the γ -ray propagation direction. We estimate that the iron moments are all within 15° of the normal to the plane of the film. These angles and the broadened spectral linewidths suggest a small distribution of the moment directions, with an overall tendency for the moments to be normal to the plane of the film. This is consistent with the observation by electron microscopy of columnar microstructures in Fe₇C₃ films deposited on glass fibers.⁵ The misfit observed at 3 mm/s seems to indicate the presence of a distribution of hyperfine fields which is correlated with the isomer shift, at least for the most intense sextet.

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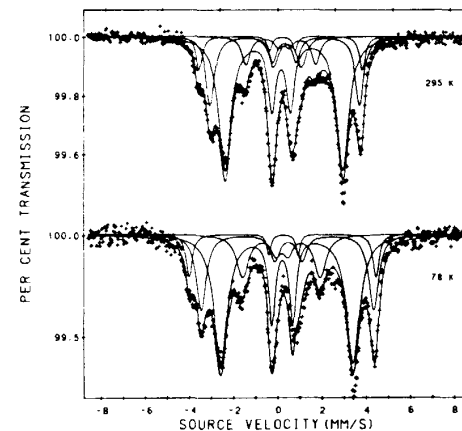


Figure 1. Mössbauer spectra and fits for Fe₇C₃ thin films, obtained at 295K and 78K.