GRAZING INCIDENCE X-RAY DIFFRACTION STUDY OF LOW CARBON STEEL CORROSION INDUCED BY CARBON DIOXIDE

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Sembiring *et al* (2000) described *ex-situ* grazing incidence x-ray diffraction (GIXRD) measurements and a parallel synchrotron radiation (GISRD) study of low carbon steel corrosion induced by CO_2, with particular reference to the transportation of natural gas from offshore deposits through mild steel pipes. In these initial studies, low-carbon steel coupons were immersed in 'formation water' at 50° C, containing CO_2 at a partial pressure of 50 psi. Samples were exposed for periods of 1 hour and 1, 3, and 7 days. The *ex-situ* GIXRD results showed the development of two phases in addition to the α-Fe phase of the mild steel substrate: (1) $FeCO_3$ (siderite) which is the product of CO_2 reacting with α-Fe; and (2) $CaCO_3$ (calcite) which appeared to have been deposited from the formation water. By contrast, the GISRD patterns showed only α-Fe and $FeCO_3$ features, with the absence of $CaCO_3$ being attributed to preferred orientation.

This *in-situ* sequel to the initial *ex-situ* paper has been conducted to observe the phases as they develop in real time, and to thereby eliminate any possibility that *ex-situ* results reflect, at least in part, phase development occurring after the steel coupons are removed from the reactor, and also to avoid contamination effects. By contrast, the *in-situ* study has primarily involved the development of a corrosion cell for use in time-resolved GIXRD experiments, to acquire phase composition data relating to the true surface structure of the corrosion films forming in solution. An *in-situ* GIXRD cell has been developed to operate with a Siemens D5000 diffractometer, such that a 6 μm layer of CO_2-laden formation water flows over the steel coupon. The water is pumped from a reservoir of formation water containing CO_2 at the required pressure. A taught Prolene film of thickness 4 μm constrains the formation water film thickness within the cell such that the impact of signal attenuation and scatter is minimal.

The paper will present *in-situ* coupon weight loss data that validate the cell design, along with our preliminary *in-situ* GIXRD results.

REFERENCE

Sembiring, S.; O'Connor, B.; Li, D.; van Riessen, A.; Buckley, C.; Low, I.; De Marco, R., *Advances in X-ray Analysis*, **2000**, *43*, 319-325.