INFLUENCE OF PREOXIDATION ON FECRALLOY EFFICACY AS A CATALYST SUPPORT

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SUMMARY

Modern techniques were used to study of the physiochemical transformations caused by thermal preoxidation of the surface of Fecralloy foil to assess the influence on coating adherence. These techniques consist of the use of a scanning electron microscope (SEM) and a laser profiling interferometer (LPI).

INTRODUCTION

- Fecralloy is the integral component of metallic monolith catalysts.

Fecralloy

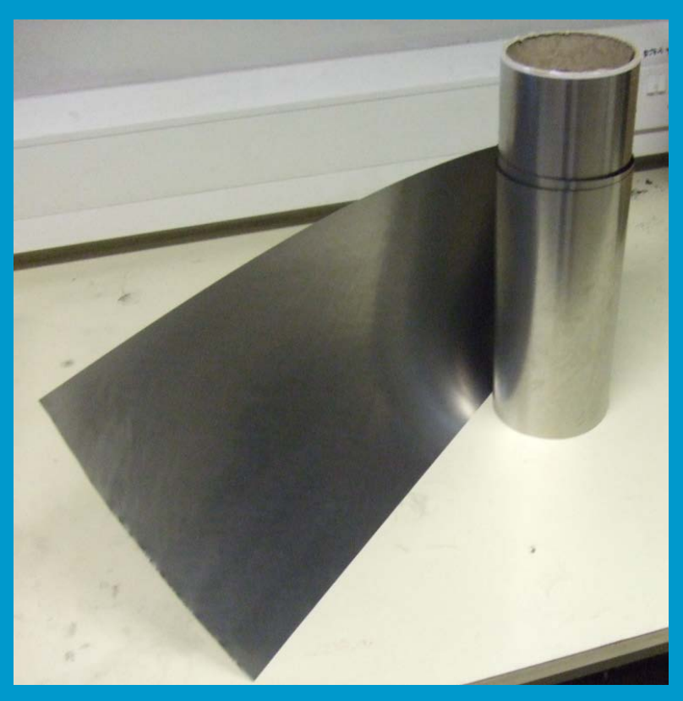
- alloy of iron, chromium and aluminium

Catalyst Support

- formed into a catalyst support having straight parallel channels

Monolith Catalyst

- coated with a PGM based slurry

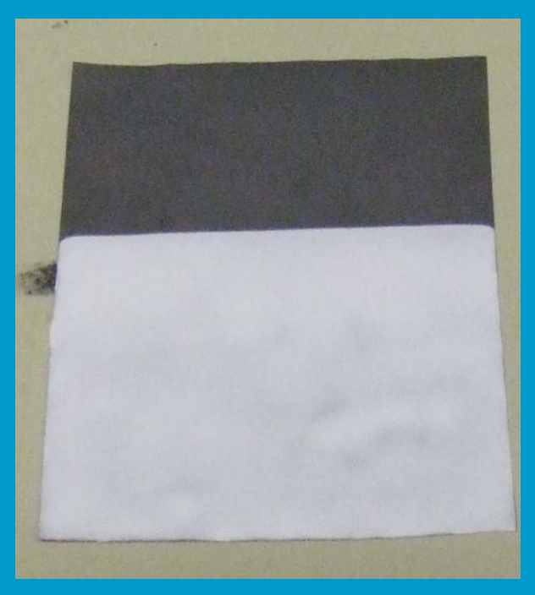


- The surface of Fecralloy foil is preoxidised to obtain the optimal microstructure onto which coatings can be properly adhered.
- The transformations caused by thermal preoxidation at 950 °C for 5, 10 and 30 h were assessed by LPI and SEM and the specific mass gained at these times was determined.
- The coating slurry ($d_{10} < 2\mu m$ and $d_{90} < 15\mu m$) is a shear-thinning suspension of γ–alumina and acetic acid which is described by the power-law model.
- An automatic film applicator is used for the coating slurry onto the Fecralloy surface.
- The coated substrate is dried at 110 °C for 1h and calcined at 500 °C for 1h.
- Coating homogeneity and thickness are assessed by SEM, while loading and adhesion are measured by quantitative weight increase and weight loss by ultrasonic vibration.

COATING USING AN AUTOMATIC FILM APPLICATOR

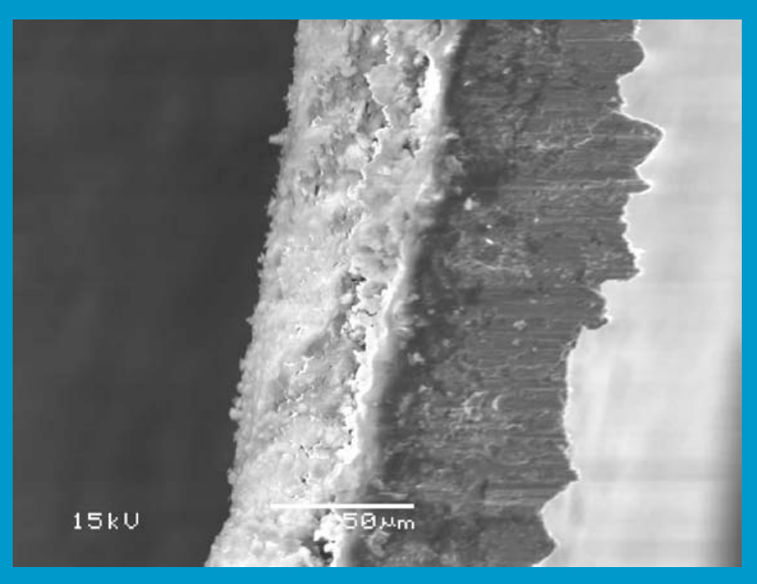
The coating slurry is deposited onto Fecralloy surface using an automatic film applicator. The resulting composite is subsequently dried and calcined.

Automatic film applicator

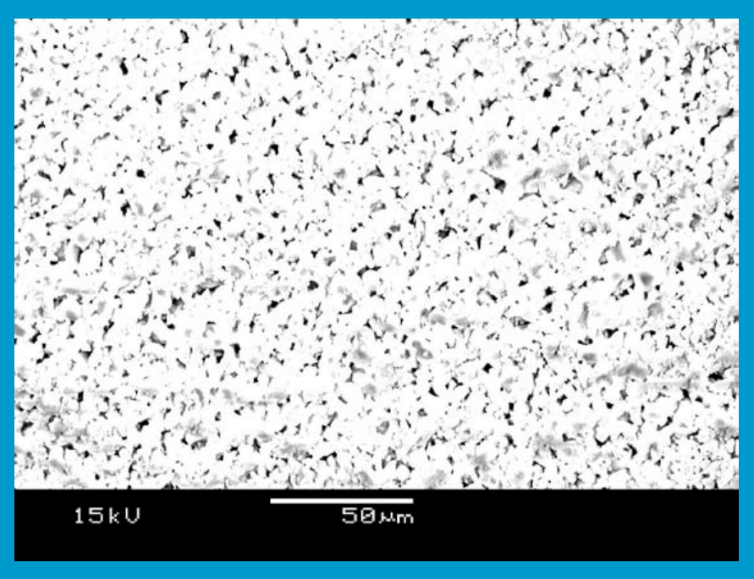


A dried and calcined product coating

Fecralloy

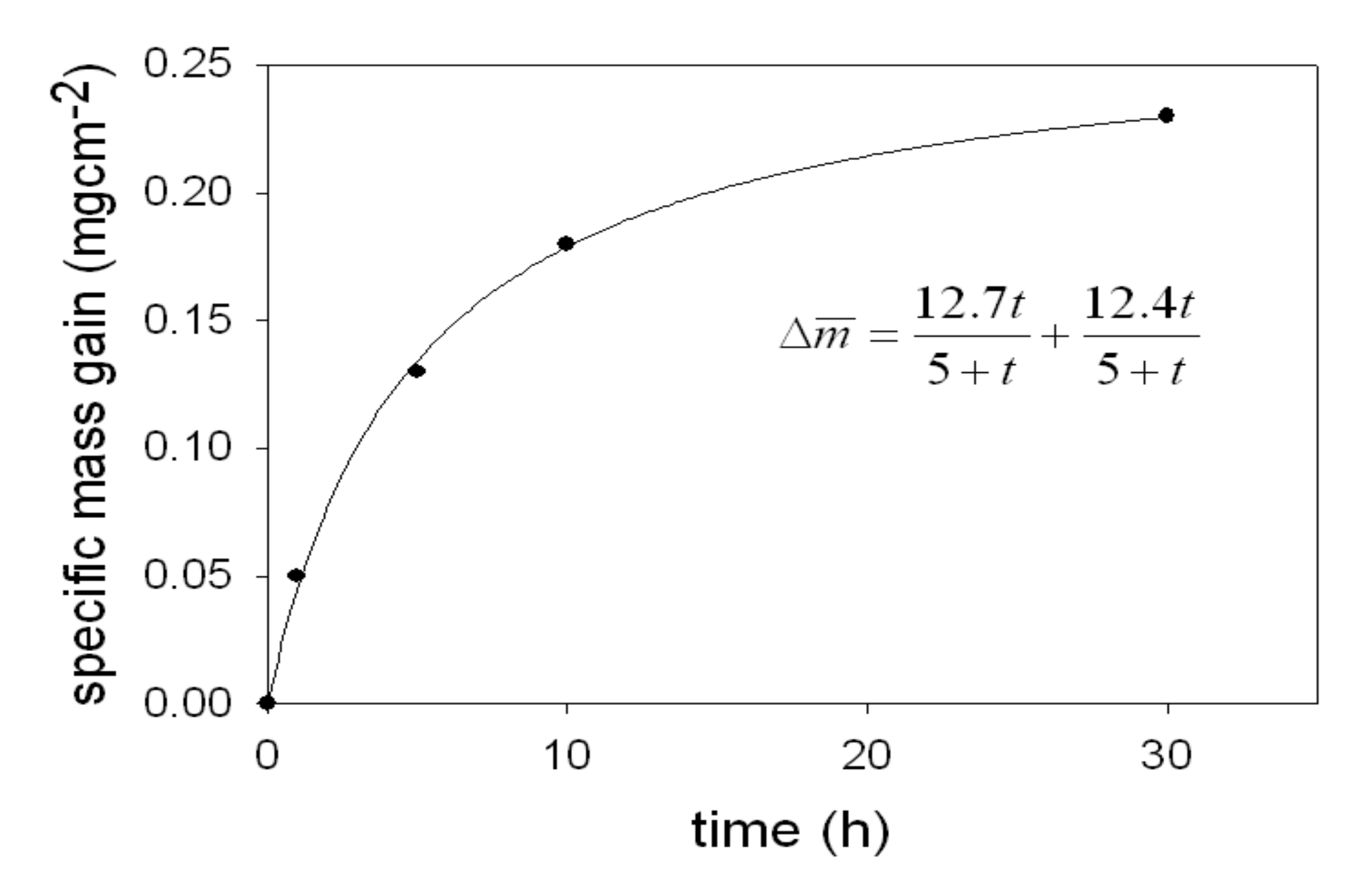


SEM picture of coating (side view)



SEM picture showing homogeneous coating (surface view)

MASS GAIN BY FOIL VS PREOXIDATION TIME



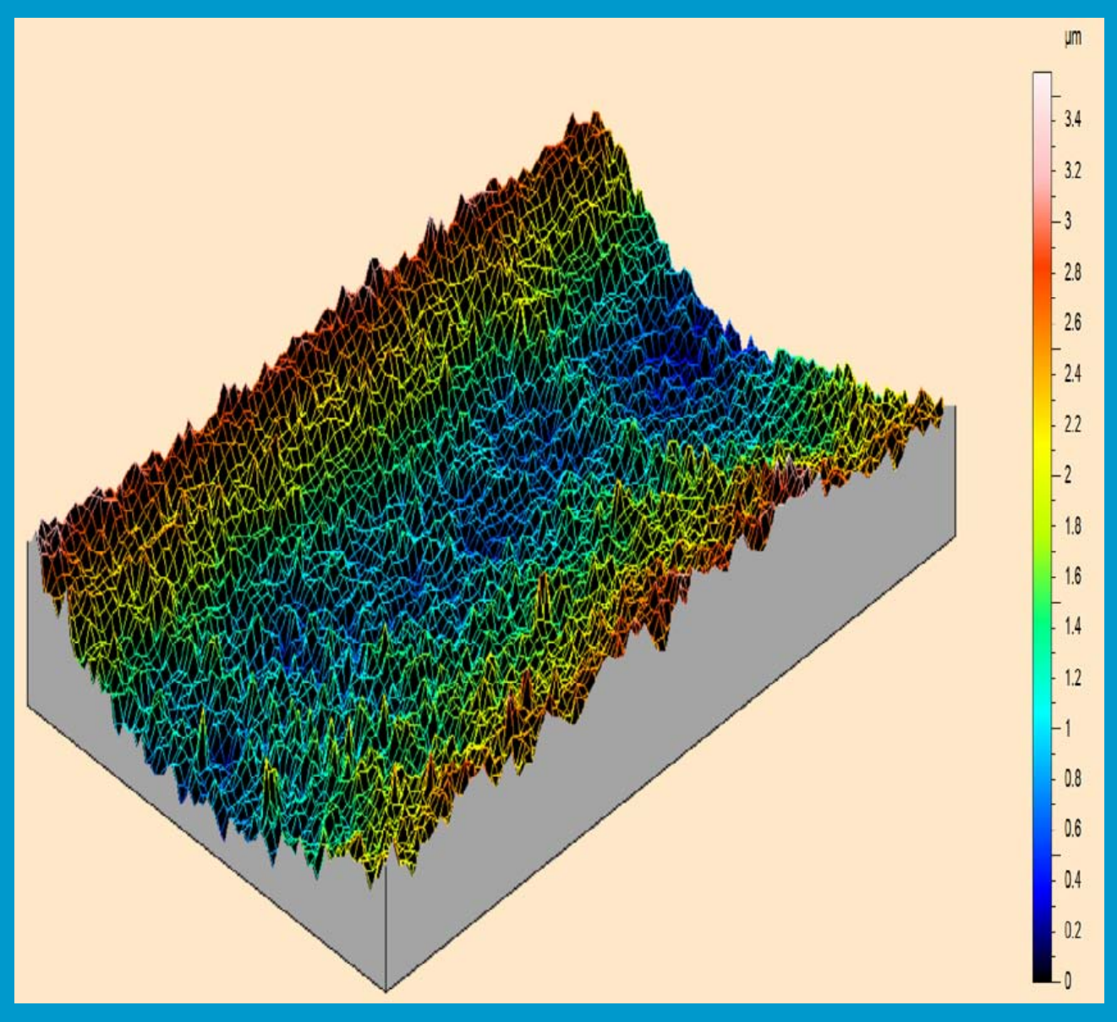
$$\Delta\overline{m} = \frac{12.7t}{5+t} + \frac{12.4t}{5+t}$$

Hyperbolic curve: specific mass of foil increased with time as oxides were formed at the surfaces.

OBTAINING THE OPTIMAL FECRALLOY SURFACE MICROSTRUCTURE

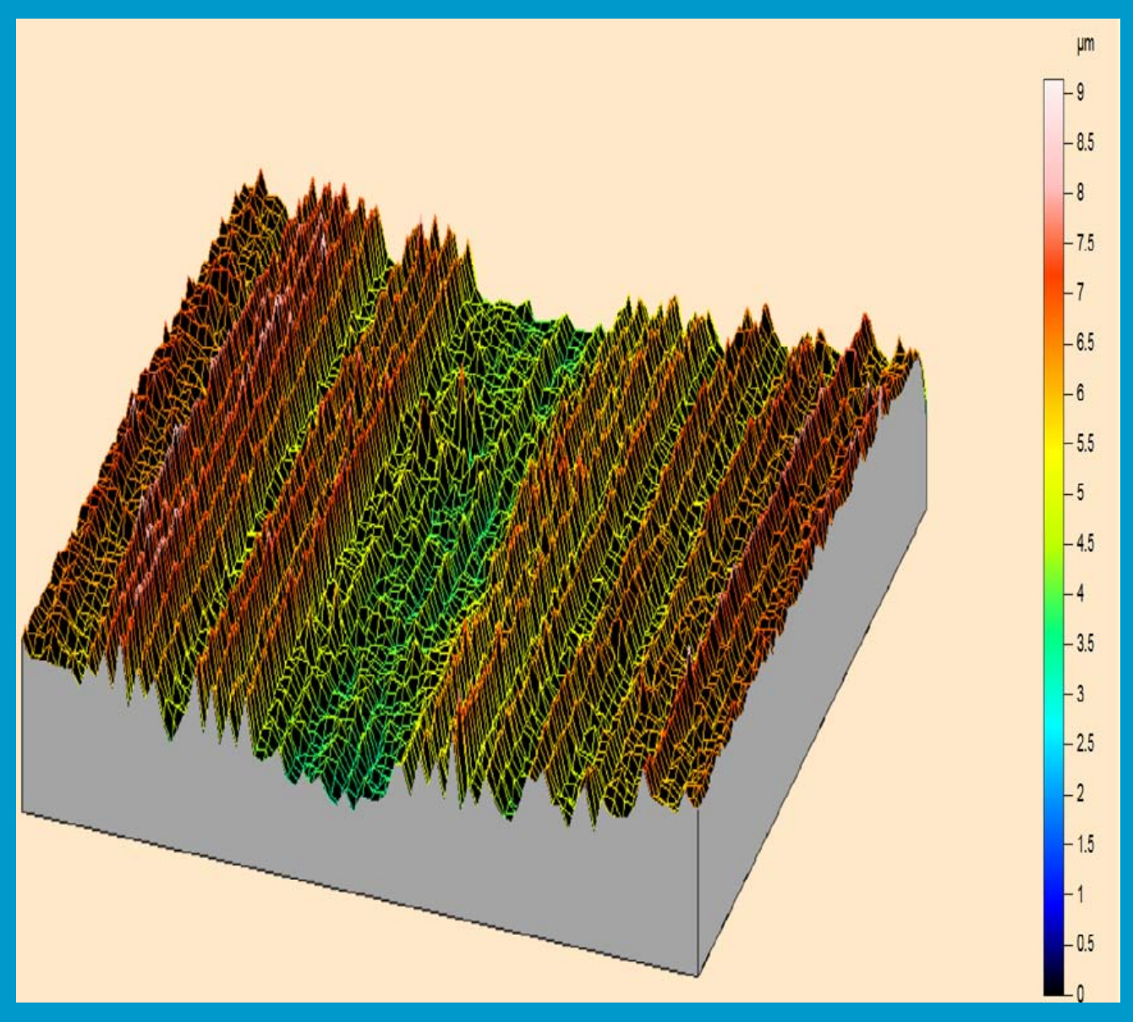
LPI – 3D ROUGNESS TOPOGRAPHY

5 h



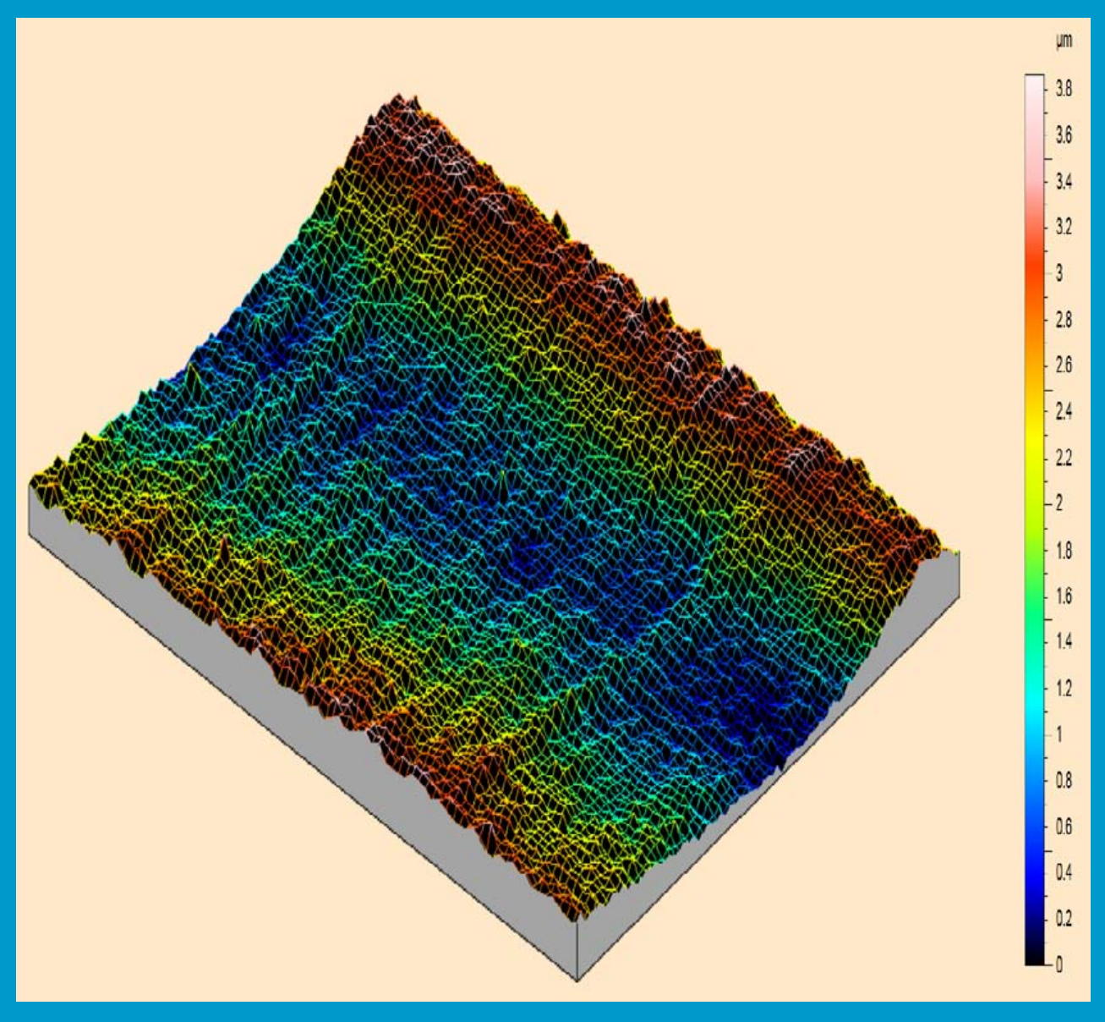
Sa = 0.35 μm, Sq = 0.42 μm

10 h



Sa = 0.83 μm, Sq = 1.01 μm

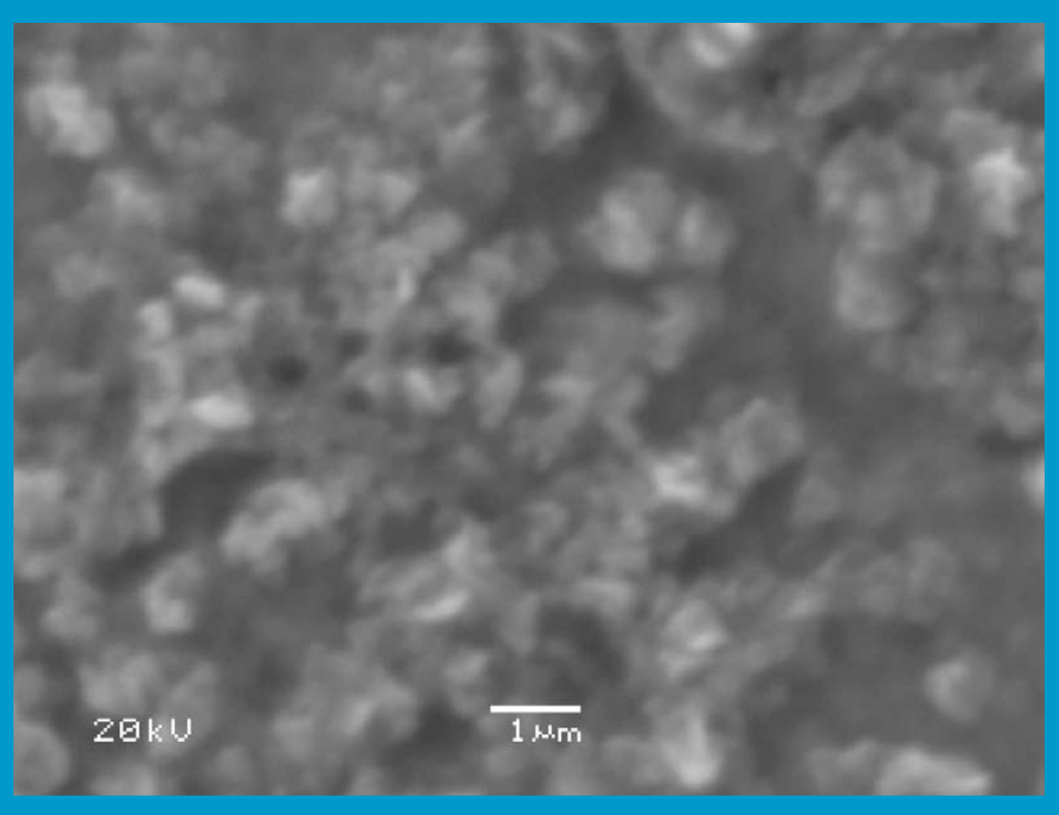
30 h



Sa = 0.52 μm, Sq = 0.69 μm

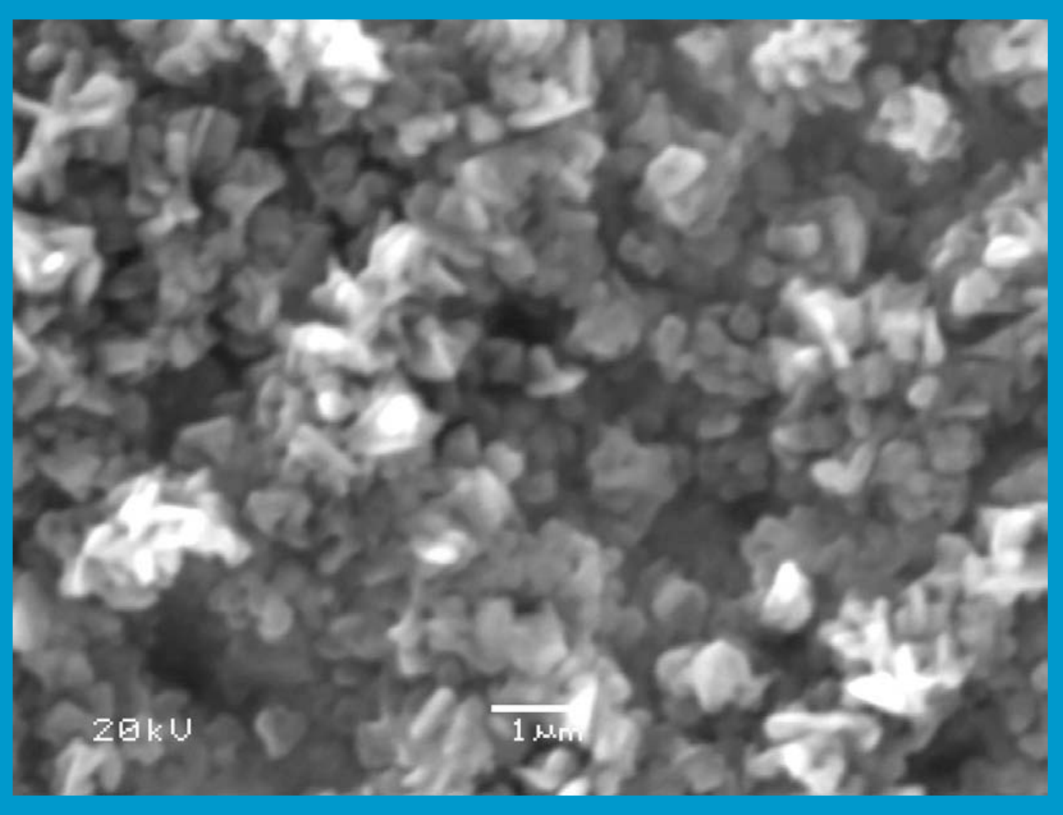
SEM – MORPHOLOGY

5 h



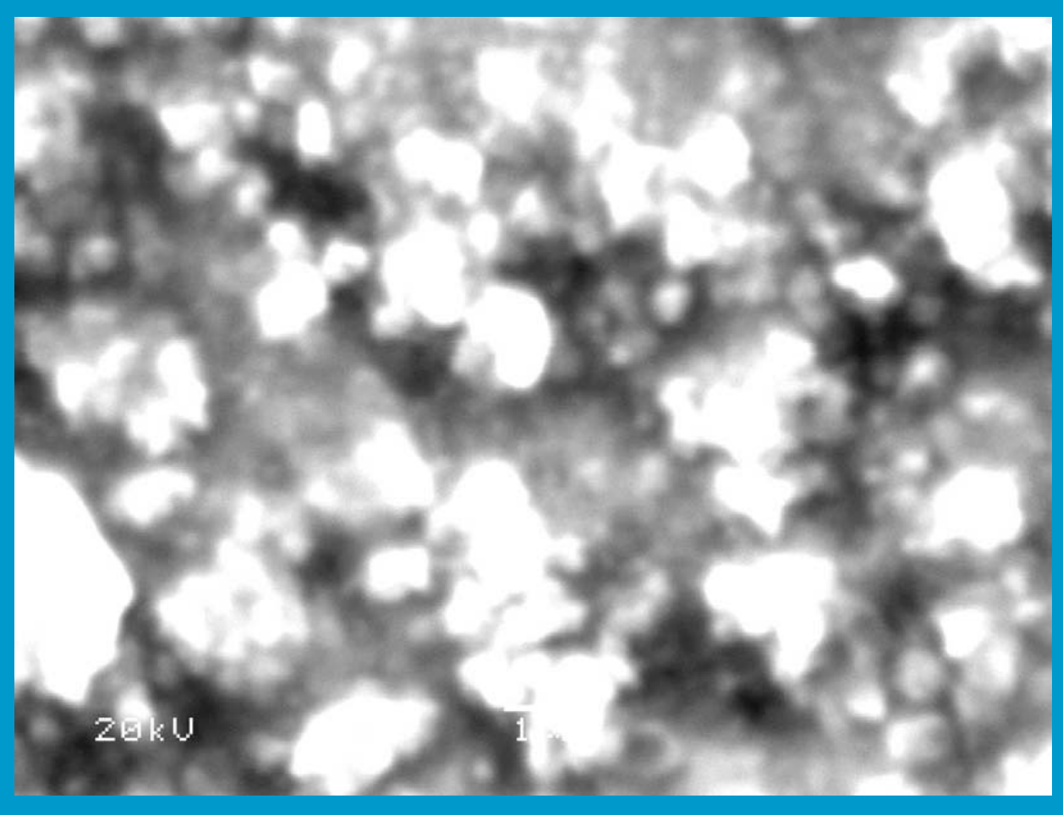
- α–alumina not properly formed, trace amounts

10 h



- conspicuous and randomly oriented α–alumina whiskers

30 h



- non uniform surface as α–alumina whiskers conglomerate

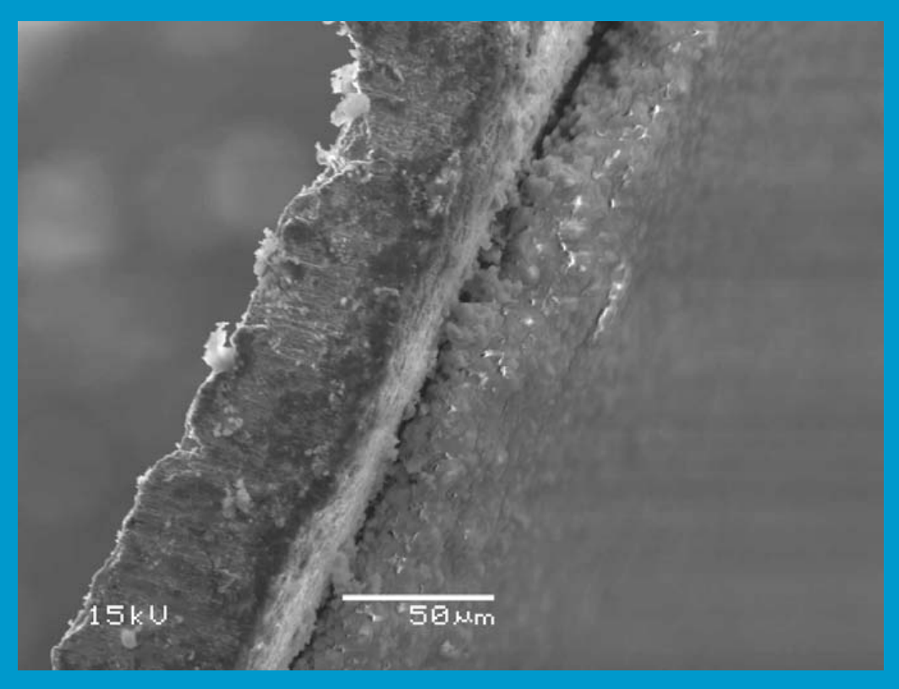
PROPERTIES OF COATINGS

preoxidation time (h)	coating loading (mass %)	mass % loss from adhesion test
5	5.2	19.4
10	7.9	9.9
30	6.8	16.3

values are averaged over 3 measurements with the standard deviation within 2%

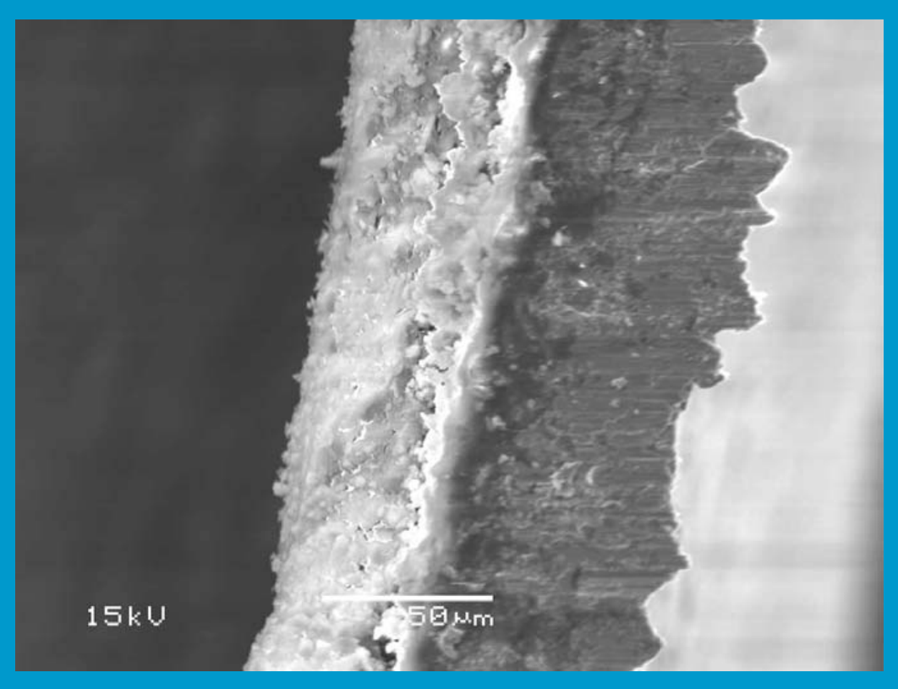
SEM IMAGES OF COATINGS

5 h



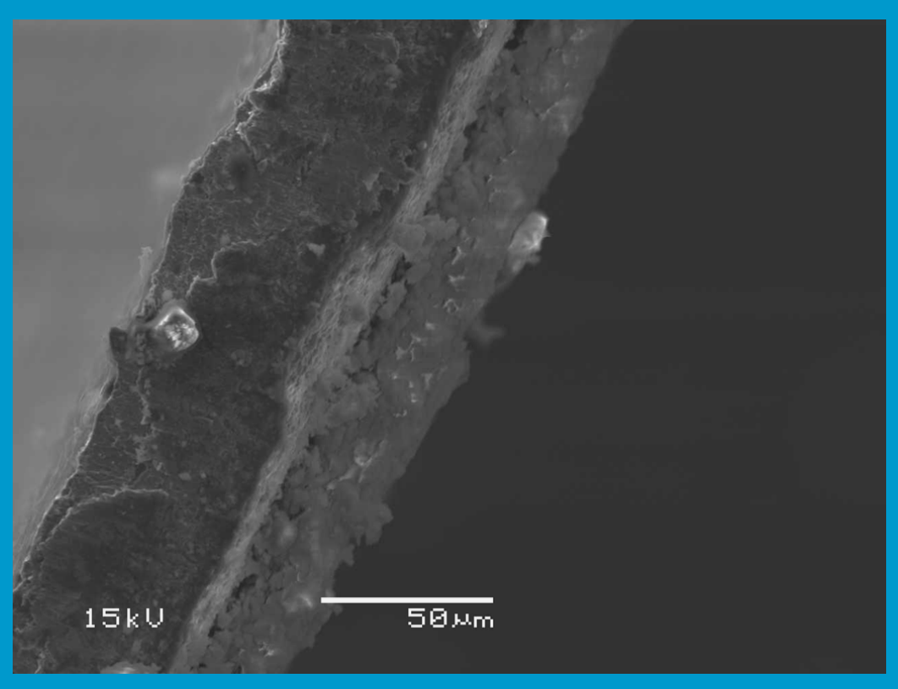
- poor adhesion as Fecralloy surface has hardly preoxidised, leading to less optimal microstructure

10 h



- good adhesion as Fecralloy has the optimal surface microstructure due to sufficient preoxidation

30 h



- prolonged Fecralloy preoxidation caused a decrease in adhesion because surface has less optimal microstructure

CONCLUSIONS

- The preoxidation conditions have been shown to be central to achieving a high degree of performance for Fecralloy as a catalyst support.
- An hyperbolic model provided the best fit for the specific mass gain of foil as a function of preoxidation time from 0 – 30 h.
- The pioneering LPI technique produced a quantitative assessment of Fecralloy roughness topography, which showed that foils preoxidised for 10 h had the highest roughness parameters (i.e. Sa and Sq).
- The SEM showed clearly the nature of α–alumina whiskers produced on the Fecralloy surface at different preoxidation times and the optimal microstructure was obtained by preoxidation for 10 h.
- The optimal coating loading (7.9 mass %) and adherence (9.9 mass %) were obtained from foils preoxidised for 10 h. Coating adherence was however poor for foils preoxidised for 5 and 30 h.

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