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Generation and testing of catalytic films on aluminium supports produced by plasma electrolytic oxidation (PEO)

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Introduction

Metastable alumina phases were obtained by thermal oxidation of FeCrAlloy rods¹ and by using plasma electrolytic oxidation method (PEO) of aluminium strips. The structured catalysts were characterised and then metal-loaded. The activity and toluene selectivity in the dehydrogenation of methyl cyclohexane was determined using a fixed bed reactor. A comparison with pelleted powder catalysts allowed evaluation of reaction pathways in this highly endothermic process.

Materials and Methods

Alumina layers of typically 75 microns thick were produced using aluminium strips (15mm x 5mm x 0.3mm, 99% purity) by PEO (Table 1) and were characterized using SEM-EDX techniques, XRD and TGA to determine morphology and adherence. The catalytic behaviour of the strips was tested after the strips were loaded with platinum (Pt) for the dehydrogenation of methyl cyclohexane (MCH) reaction.

Table 1. PEO process conditions²

Material	Electrolyte	Current density	Frequency	-ve to +ve Pulse Ratio	Oxidizing time
Al strip (1.5*0.5cm)	2.8g/L KOH 10.5g/L NaSiO ₃	500 mA/cm ²	50Hz	1.3	30min

Results and Discussion

The alumina layer was shown to be well anchored and the back-scattered SEM image showed the multi-faceted porous outer oxide layer (Fig 1a) with the elemental mapping confirming the presence of a predominantly Al₂O₃ layer approximately 75 microns thick (Fig 1b). XRD confirmed the presence of aluminosilicate (Mullite) and a range of alumina phases from alpha to gamma (Fig 2).

The hierarchical structured catalysts (typically 1g of Al₂O₃) were tested for dehydrogenation of methylcyclohexane (MCH) in a fixed bed reactor. To obtain a detailed quantitative assessment of the selectivity toward the formation of toluene at various operating conditions, all samples were analysed in a GC-MS (Agilent Technologies, Model-6890N) equipped with a HP-5MS capillary column (50 m x 0.25 mm i.d., 5 % Phenyl and 95 % methylpolysiloxane). High conversions (50% - 99 %) of MCH and increased selectivity (>98%) towards the formation of toluene were achieved at, 340 °C – 400 °C. Owing to the endothermic nature of the reaction there was an initial temperature drop of 10 °C – 15 °C at all reaction temperatures whereas pelleted 1 wt. % Pt/Al₂O₃ for the same reaction conditions resulted in a temperature drop typically of 30 °C – 70 °C.

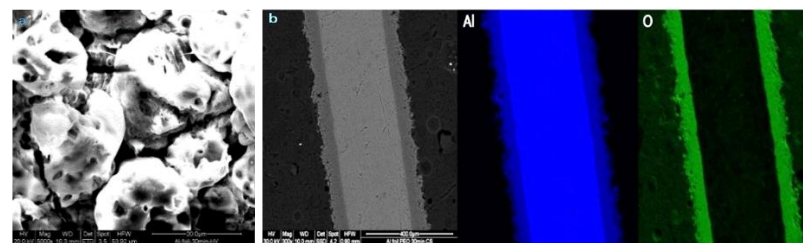


Figure 1. SEM image of the mixed phase PEO generated Al₂O₃ layer (a) and, EDS elemental mapping of a polished cross-section (b).

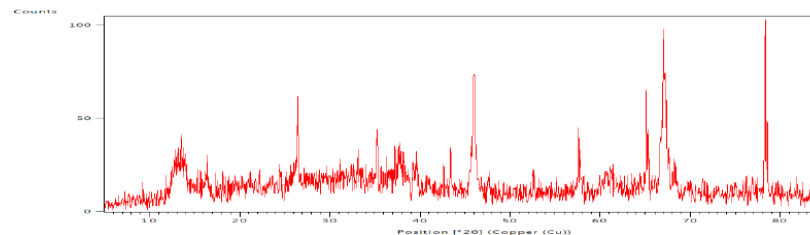


Figure 2. XRD spectra of mixed phase Al₂O₃ strip synthesised by PEO

Long term life tests were performed at T=400 °C and W/F=14690 g s mol⁻¹ on the developed catalysts and compared with a conventional commercial pelleted 1 wt.% Pt/Al₂O₃ catalyst (supplied by Sigma-Aldrich Figure 3 describes the change in conversion over a period of 400 h for the structured and pelleted catalysts. The alumina coating was adherent post catalytic testing.

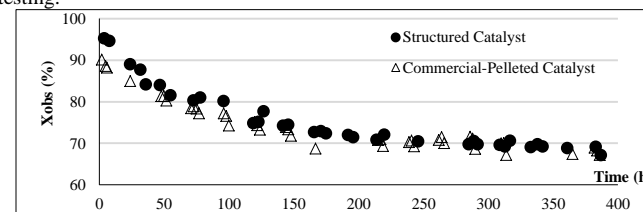


Figure 3. Long term life tests of 1wt % Pt/Al₂O₃ on supported catalysts compared with conventional pelleted catalysts at 400 °C and W/F=14690 g s mol⁻¹ describing change in catalytic activity with time

Significance

Stable hierarchical structured catalysts showed comparable high conversion and selectivity for MCH dehydrogenation with improved heat transfer properties.

References

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