Industrial process problem solving, routine analysis consultancy and materials analysis.



Thermal Production Process Simplification and Catalytic Activity Improvement for a Ni-Cr Catalytic Bed Using a GC Microreactor and XPS

Production Process Improvement Issue

The methane catalytic gas cracking capability of a simple nickel-chromium catalytic bed is known to be sensitive to the thermal history of this manufactured component. The production process historically involved a complex and expensive series of thermal processes and it was thought that the production process could be simplified without risk to the product's effectiveness.

А microreactor coupled to a GC (Gas Chromatograph) as well as XPS (X-ray Photoelectron Spectroscopy) were used to evaluate and explain why the methane cracking varied with thermal processing. Ultimately the aim recommend the most was to effective combinations of thermal treatments for the catalyst, with the target of process simplification.

Catalytic Activity Measurements and Surface Chemistry

The catalytic GC-microreactor shown in fig.1. was constructed in order to determine the methane cracking ability of the components.

The catalytic reaction was modelled on the steam reforming reaction in which methane in the presence of water reacts to form carbon monoxide and hydrogen.

 $CH_4 + H_2O \rightarrow CO + 3H_2$



Fig.1. CG and catalytic microreactor



Fig.2. XPS instrument used in to explore surface chemistry.

The catalytic activity of the components were found by GC comparing the amount of gases present after passing the gas over the bed of components in and out of the hot zone of the furnace. As the catalyst is only active at high temperature, a quantitative efficiency could be determined across a range of candidate thermal treatments.

XPS is a technique which chemically analyses the top few atomic layers of a material. It not only permits the surface elements to be determined, but also the chemical state and atomic concentration (directly from the areas under the peaks). As the catalytic process occurs at the surfaces of materials, XPS was used to characterise the candidate processed product before and after GCmicroreactor testing.



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XPS and CG-Microreactor Results

Fig.3. Example XPS results for two catalytic components following different production thermal treatments. Note the clear Ni $2p_{1/2}$ and $2p_{3/2}$ peaks for the Non redox stoved components (pre microreactor) and the high catalytic activity.

The GC-microreactor results showed which processing steps produced the best catalytic response.

The surface chemical compositions of the product derived from XPS spectra (similar to those shown in Fig.3) were correlated with the catalytic activity measured in the GC-microreactor. One such plot is shown in fig.4.

XPS confirmed that the removal of the redox process increased the amount of surface metallic nickel to the greatest extent and increased the component's catalytic activity most significantly.



Fig.4. The atomic concentration Ni/Cr ratios measured by XPS after testing in the GCmicroreactor is linked directly to the catalytic activity for cracking methane. The more surface nickel the better.

Process Improvement Solution

Gas Chromatograph work in a laboratory microreactor was able to determine the most effective combination of thermal processes to produce the best methane-cracking catalyst bed.

The chosen combination of thermal processes were able to reduce the number of necessary production process steps and reduce the manufacturing cost. This was possible with an increase in catalyst product performance.

The surface compositions derived from XPS made it possible to explain why this improvement was seen. It was a result of an increase in surface metallic nickel.

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> LPD Lab Services Phone: +44 (0)1254 676074 E-mail: enquiries@lpdlabservices.co.uk Web: www.lpdlabservices.co.uk