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Guest Editorial

The Importance of Characterisation Techniques

We have previously described some of Johnson Matthey's core competencies in modelling (1) and the control of advanced materials at the atomic scale (2). The third of these competencies, and a vital component of the company's strategy to develop high performance solutions to its customers' problems, is characterisation of materials.

Materials characterisation is a huge and diverse field. One place perhaps to start is at the beginning, and the first principle to consider is the depth to which characterisation supports new materials discovery:

- Measuring a material's property allows experimental improvement in the property;
- Making unique measurements allows differentiation by improvement in unique areas;
- Insight into compositional and structural origins of material properties enables rationally designed improvement.

Taking a model heterogeneous catalyst, of a metal supported on an oxide support (such as platinum on alumina), hydrogen chemisorption allows for empirical exploration of preparation strategies leading to increased surface area. As well as the support it is essential to control Pt content between preparations to ensure commercial efficiency, thus even for a relatively simple material the number of measurements needed to achieve empirical success increases rapidly.

The "unique" measurements are measurement capabilities developed for a bespoke solution to a particular question or performed with instrumentation or facilities which have a high barrier to access. Returning to the model material, the optimum metal surface area available depends upon nanoparticulate Pt dispersed over the alumina surface. Transmission electron microscopy (TEM) coupled with automated image processing can count and describe particle size, shape and dispersion. TEM is not a unique technique, yet developed know-how in sample preparation, data collection, image processing and data interpretation varies widely between practitioners. Reliable access to material properties such as particle size distribution at nanoscale is key to enabling empirical optimisation and opens the potential for control of both size and distribution.

Within the third category, the delivery of insight requires an internal connection between a compositional or structural property and the behaviour of a material. Hence most "*in situ*" and "*operando*" approaches sit within this area. Taking the Pt on alumina model further, observation of nanoparticle sintering in response to changing temperature can be achieved with commercially available equipment. Direct observation conducted with a time capture component yields significantly more insight than empirical approaches.

There is therefore another level in the characterisation hierarchy to pursue:

- Making measurements that yield temporally limited structure (reactive intermediates), kinetic or energetic values during *operando* studies enables the *in silico* modelling of materials and the prediction of performance based on proposed materials structure.

There is much fertile research ground to explore in the gap between current modelling capability (1) and a full description of a material integrated across length scales.

Characterisation Driving Innovation

The above pressures would tend to suggest that characterisation demand will continually drive towards the delivery of the ultimate, since the richness of the information has a direct impact on the ease with which materials discovery can be accomplished. The predictable trade-off is with cost and time.

Figure 1 describes the dilemma. At the pinnacle of the triangle is the delivery of intellectual property (IP) in materials development. Empiricism (discovery through systematic exploration of the preparation variables) is furthest away from IP and requires significantly more (albeit lower complexity) characterisation support. It is no surprise that, as characterisation complexity increases, so does the stimulation towards insight and designed improvement, hence one moves closer to the delivery of IP with fewer measurements. But cost increases and the availability or throughput drops.

Not implicit in the understanding of this model is the bias from which it is observed. One challenging thought is that "characterisation" has traditionally operated as a service activity to the synthetic researcher: i.e. people make materials, then analyse, activity test and try to work out what the results mean to formulate the next experiment. This "perspiration approach" requires a huge bias in delivery of capability in order to drive innovation. Generally speaking, more of the picture of a material is needed before the further stage can be formulated and hence the demand for measurement provision is exponential. The exponent is exacerbated by materials complexity, which increases with the need to achieve new performance requirements. The risk is that a gap between capacity to make materials, and the capacity for characterisation, limits the rate of improvement.

The alternative turns this on its head: model-led characterisation demands materials to confirm hypotheses, enabling predictive optimisation. Fewer materials are synthesised yet insightful measurement stimulates the inventive step (the "inspiration approach"). More research support is committed to development of characterisation methods which are consistent with insight delivery for complex materials.

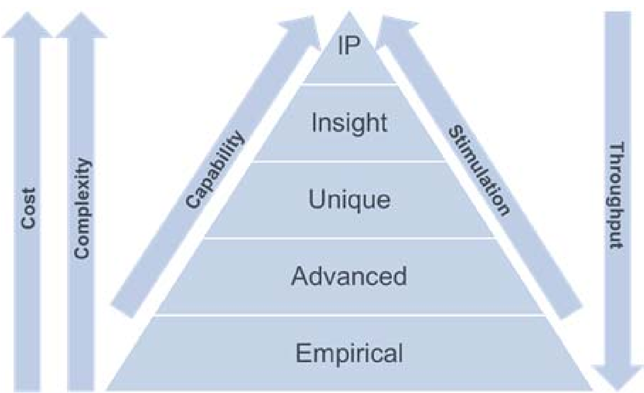

Fig. 1. Innovation through characterisation

Rewarding Collaborations

We already see this happening. Increasingly, academic and industrial researchers are either demanding or beginning to develop characterisation methodologies to support solutions to their problems. The market is shifting to meet the arising need. Characterisation development is a rewarding area for collaborations: the intellectual challenge to extract predictive information from materials is high, and has high impact, yet the exploitation of insightful methods can be separated from commercial materials development through use of the simple yet realistic model systems which are needed to drive the cutting edge.

One area where this is already yielding a rich collaboration is the rapidly developing partnerships centred at national research facilities. In Johnson Matthey's case, the Harwell campus in the UK incorporating Diamond Light Source, ISIS the neutron source and the Lasers for Science facilities all have capabilities to enhance characterisation development. The UK Catalysis Hub consortium is also on campus and is developing researchers with the capacity to develop characterisation methodologies. Working in this environment, providing rich problems and collaboration support opens up great promise for the development of future solutions to the four objectives for characterisation.

So here we should celebrate this issue based on characterisation methods. The thread that binds them is the development of methodologies seeking to acquire information about increasingly complex systems, towards finding answers to critical problems for real-world applications.

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