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Aberration Correction in Dynamic In-situ Studies of Nanoparticles

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Aberration correction has become well established for main stream high resolution electron microscopy both in TEM and STEM [1, 2] and there are significant additional advantages in dynamic in-situ TEM, where the effects are truly transformational. This is because in that mode there is usually the opportunity to acquire only a single discrete image from each field of view; precluding interpretational techniques based on acquiring through focal series of images of an assured static scene (and this may be a more general problem too?). The improvements in the quality and veracity of structure imaging in the inter-atomic spacing range of 0.3 to 0.1nm is at least as important as the resolution extension to below 1 Angstrom; with the latter being maintained in hot stage operations to 500C and above [3, 4]. Our work on the structure, stability and properties of nanoparticles, with especial relevance to heterogeneous catalysis applications, benefits from aberration correction in several ways:

- Operation at close to zero defocus (compared to an optimum of -61nm for the uncorrected instrument), reducing considerably image delocalization effects which are especially important at structural discontinuities such as defects and internal and external surface interfaces which dominate properties, and simple sizing up. Fig.1(a) is an example of a classical form of multiply twinned gold nanoparticle 7nm in size showing the pie shaped internal structure and consequent modification of the surfaces presented externally. Fig. 1(b) shows a smaller nanoparticle, in this case a single crystal, of a type where the properties are reported to be strongly size range dependent with high chemical activity, even for gold [5].
- Avoidance of oscillations in the CTF with their missing pass bands and contrast reversals with spatial frequency and operating defocus, as shown in Fig.2, in comparison to the uncorrected instrument [4].
- Greater HRP polepiece gap of 4.3mm to accommodate a regular hot stage and other in-situ apparatus without incurring the usual performance penalty in an uncorrected system, assisted by the small (~20%) and in practice unimportant change in Cc in this gap range from the UHR lens. The favourable Cc effect does not extend in the same way to much wider gap lenses.

Our microscope also has a number of other configuration changes for purpose; most notably the use of TMPs with oil-free dry backing pumps, instead of ion and diffusion pumps, for the main column vacuum systems, to provide gas tolerance for hot stage out gassing and for future gas injection ETEM developments. Complementary chemical analyses are provided with an in-column Omega EELS and EDXS between the polepieces. It is also controlled remotely with the operators, power supplies, mechanical pumps and ancillary systems all in different rooms to the main column unit in its isolated pod. The planned development of the instrument includes adding to the present controlled high temperature regime facilities to provide a controlled gas environment around the sample for the study of reaction mechanisms and key

intermediate phases which may be metastable with respect to temperature or atmosphere but are important in reaction processes. These may be key to understanding the fundamental basis of reactions but they are generally inaccessible through post reaction studies extracted from the reaction sequence conditions of atmosphere and temperature.

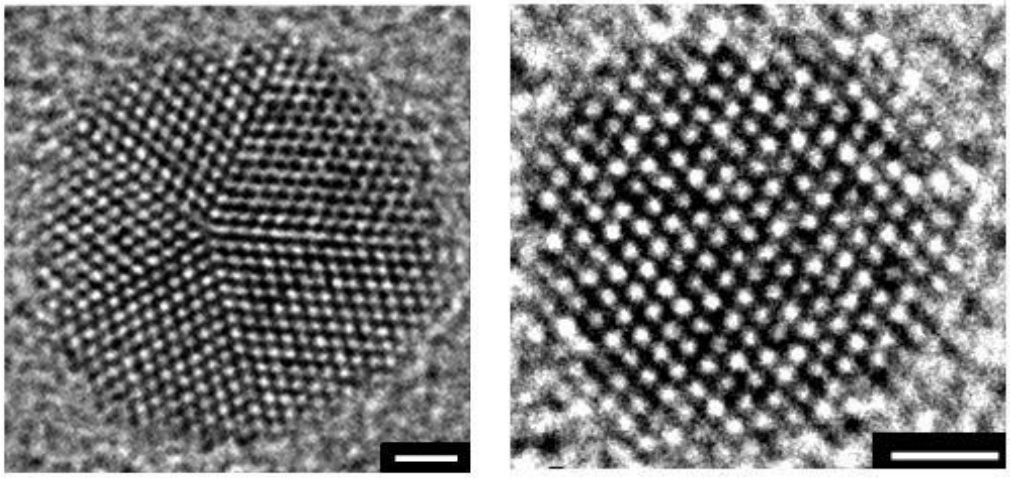

Fig. 1: Aberration corrected TEM images of gold nanoparticles with a classic multiple twin structure in (a) at a size of 7nm, where it behaves catalytically pretty much as bulk gold, and a single crystal in the size range of 2-4nm where it is expected to behave with high oxidation activity, e.g. for CO conversion. Scale markers are 1nm

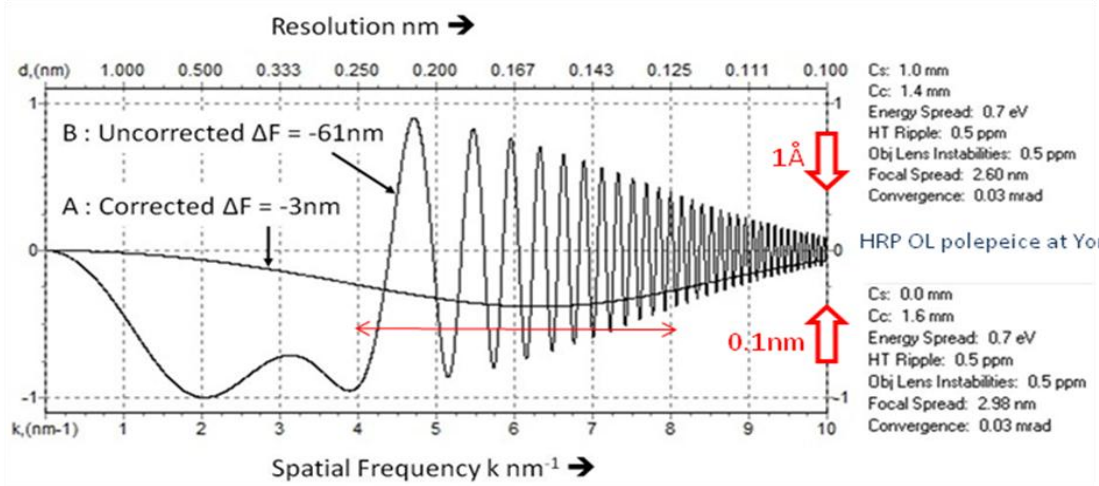

Fig. 2 Contrast transfer function calculated for the corrected instrument (A) and for the uncorrected native York 200kV microscope (B).

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