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Introduction: Characterisation of ceramics

Within the past decade there have been significant additions to available experimental techniques for characterisation of ceramic structure or composition and also in the level of sophistication of older-established techniques. Our knowledge of mechanisms underlying the processing and performance of important classes of structural and functional ceramics has also reached an advanced state via application of a wide range of imaging, diffraction and spectroscopic techniques. Hence it is an appropriate time to survey the current 'state of the art' in such techniques. The theme of this series of papers is that of ceramic characterisation over a range of dimension, down to the atomic scale. The papers have been selected to review the key microscopies and spectroscopies, illustrated either with case-studies of recent research on important ceramic systems or with more extensive reviews of representative classes of ceramic microstructure.

It is now over 30 years since the advent of ion-beam specimen preparation and the first application of moderate-resolution transmission-electron microscopy (TEM) to the early silicon nitride based ceramics. In the early 1970's TEM imaging and diffraction at Warwick University provided conclusive evidence for the role of additives in liquid phase sintering and the constitution of grain boundary residual phases. At that time X-ray spectroscopy (EDS) at high spatial resolution was not available such that Auger spectroscopy of intergranular fracture surfaces was necessary to identify grain boundary composition. Using the current range of field-emission microscopes (FEG TEM), imaging, X-ray spectroscopy and electron energy-loss spectroscopy is attainable on the nm scale. These techniques are described in the review of microstructure in Si-based ceramics (Paper 1).

In the era of 'advanced' structural ceramics, exemplified by silicon nitrides or zirconias, there has been a tendency to overlook the considerable developments in higher-performance technical ceramics and refractories, which have much greater volume-application. The microstructures of refractories are surveyed in Paper 2, illustrating the use of optical, electron and cathodoluminescence imaging.

Oxide ceramics (aluminas and spinels) are the focus of Papers 3 and 4. Property increments, especially in aluminas, have been obtained from the ability to fabricate theoretically-dense, sub-micron grain-size ceramics using colloidal precursors and novel sintering conditions (sinter-HIP or spark-plasma sintering) or the use of grain-boundary segregant ions. The characterisation of crystal orientation, grain boundary type and segregation level is exemplified for optically transparent oxides, in Paper 3, using electron back-scatter diffraction (EBSD), high angle dark-field imaging (HADF) and atomic-force microscopy (AFM). The main application area for alumina ceramics remains that of tribological components and the mechanisms underlying wear are reviewed, with reference to surface microscopies, in Paper 4.

Papers 5 and 6 provide examples of the considerable progress in analytical TEM applied to functional oxide ceramics. Paper 5 focuses on the use of EELS, with special reference to fine spectral structure (ELNES) combined with surface-sensitive electron spectroscopy (XPS), to resolve specific problems in a Ti-Cr oxide for gas-sensing application. Paper 6 exemplifies the recent progress in quantitative X-ray analysis of light elements when substantial absorption corrections are necessary for the EDS spectral data from an oxide ceramic containing heavy elements (Pb and Nb).

Nuclear magnetic resonance (NMR) spectroscopy is now established as a key technique for local structure determination of both crystalline and non-crystalline ceramics. The recent access to very high magnetic fields and isotopic enrichment of previously difficult nuclei has expanded its application to a broad range of both functional and structural ceramics. This technique is illustrated in Paper 7 with reference to non-crystalline Ti-O ceramics which have numerous applications in catalysis and optics. This paper, in particular, emphasises the need for a multi-technique approach to complete characterisation, combining magic angle spinning NMR (MASNMR) with infra-red (FTIR) spectroscopy and Neutron scattering. A more extensive review of neutron-beam techniques applied to a range of ceramic systems is provided in Paper 8.

Paper 9 describes the use of micro-Raman spectroscopy to provide not only spatially-resolved structural and compositional ceramic data but also to quantify the state of residual or applied stress, illustrated here for non-stoichiometric SiC monofilaments.

The final paper is based on acoustic imaging, using unconventional excitation of the acoustic waves with an electron beam in a scanning electron microscope (SEAM), thus providing evidence for sub-surface microcracking within hard ceramic coatings but the technique has generic application to varied surface-engineering phenomena.

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