



INTERNATIONAL ATOMIC ENERGY AGENCY  
UNITED NATIONS EDUCATIONAL, SCIENTIFIC AND CULTURAL ORGANIZATION



# INTERNATIONAL CENTRE FOR THEORETICAL PHYSICS

34100 TRIESTE (ITALY) - P.O.B. 586 - MIRAMARE - STRADA COSTIERA 11 - TELEPHONE: 2240-1  
CABLE: CENTRATOM - TELEX 460892 - I

SMR/388 - 4

## SPRING COLLEGE IN MATERIALS SCIENCE ON "CERAMICS AND COMPOSITE MATERIALS" (17 April - 26 May 1989)

---

CERAMICS TO BE JOINED 10 YEARS FROM NOW

M.H. LEWIS  
Department of Physics  
Centre for Advanced Materials Technology  
University of Warwick  
Coventry CV4 7AL  
UK

---

These are preliminary lecture notes, intended only for distribution to participants.

M H Lewis

Centre for Advanced Materials Technology  
University of Warwick

ABSTRACT

Monolithic ceramics containing  $\text{Si}_3\text{N}_4$  or  $\text{SiC}$ , developed during the 1970s, are likely to remain the major contenders for high temperature applications, at low stress levels, well into the next decade. The more recent evolution of  $\text{ZrO}_2$ -toughened ceramics represents an important step in engineering design potential but the toughening mechanisms are confined to low temperatures. The offer of 'damage-tolerant' ceramic-matrix composites, avoiding the problems of brittle fracture mechanics, has initiated intense activity in their fabrication over the past few years. The difficult and expensive fabrication, requirement for non-reactive interfaces and high-temperature instability dictate that their widespread application is unlikely within the next five years. A solution to some of these problems is anticipated in this period, such that composites will adopt a progressively larger part of the market in 10 years. Bonding of such high-strength microstructurally-heterogeneous materials will amplify many of the existing unsolved problems in monolithic ceramic joining.

1. INTRODUCTION

The past decade may be viewed as the dawn of a new era in the development and application of ceramic materials as structural, electronic, magnetic or optical components. 'Advanced' or 'High-Technology' ceramics are most frequently associated with engineering applications, from cutting tools to heat-engine components, but there are rapid growth areas for electro-ceramics from integrated-circuit substrates and packaging to the new superconducting materials. World sales for advanced ceramics have risen by a factor of ten since 1977 and is predicted to increase from the current - \$6,000 million to more than \$25,000 million in the next decade. A large part of this figure is due to electronic applications, but with a solution to the ceramic 'toughening' problem via. development of composites the structural market will be proportionately increased.

A broad classification for advanced ceramics, in terms of their properties, applications and microstructures is presented in Fig.1. The main subject of this survey is that of 'structural' ceramics, based on their exceptional mechanical or thermo-mechanical properties. The 'Si-based' ( $\text{Si}_3\text{N}_4$  and  $\text{SiC}$ ) monolithics are important developments of the 1970s, followed by the  $\text{ZrO}_2$  'toughened' ceramics in the latter part of the decade. In the 1980s there is intense activity in development of ceramic-matrix composites, designed principally to enhance toughness, with early applications for the more easily fabricated whisker or dispersion-toughened microstructures. Although the current publicity surrounding R & D in ceramic composites suggests that they will dominate the structural-ceramics market in the next decade, there are widespread problems concerning fabrication, high temperature stability, resistance to cyclic and multiaxial stresses together with economic factors which dictate that monolithics will retain their position in lower cost/lower stress structural applications as well as in the electro-ceramic sector. For this reason it is appropriate to review the current status of monolithic ceramic development, which will continue to advance, mainly via. refinement of existing materials, into the next decade. This will be followed by a survey of ceramic matrix composites, identifying those problems of fabrication, microstructure and component availability which are far from solution. Finally a prediction is made of problems to be solved in the bonding of such chemically and structurally heterogeneous materials.

## 2. MONOLITHIC STRUCTURAL CERAMICS

### 2.1 Microstructural types and fabrication routes.

Monolithic ceramics are normally fabricated by the densification of a porous particle aggregate - a process known generically as 'sintering'. The particular chemistry and sintering mechanism dictates the type of microstructure in the ceramic component (Fig.2). For example, monophase silicon carbides result from pressureless solid-state sintering of ultra fine particles containing surface-active sintering catalysts, boron + carbon, the latter being a surface de-oxidising agent and the role of boron believed to be in modifying surface energy or diffusivity. Monophase  $ZrO_2$  ceramics may be similarly produced in a fine-grained tetragonal-crystal form (TZP) from high purity powders containing ~ 3%  $Y_2O_3$  as a crystal-stabilising additive which also influences diffusivity (Fig.3,4). A totally solid-state sintering mechanism is rare and in many systems the inadequacy of diffusion kinetics below decomposition temperatures necessitates catalysis via a liquid phase. This is true for the wide range of silicon nitride ( $Si_3N_4$ )-based ceramics, including the 'Sialons' [1]. In these materials the liquid transport medium is a silicate, derived from reaction between metallic-oxide additives (e.g.  $Y_2O_3$ ,  $Al_2O_3$ ) and impurity surface  $SiO_2$ . In rare cases the liquid is 'transient' due to solid-solution of its components in the major phase, resulting in near-monophase microstructures (e.g.  $\beta$ 'Si-Al-O-N and  $\beta$ 'Si-Be-O-N) - Fig.2. Most frequently the liquid remains as a glassy intercrystalline silicate in a diphasic ceramic (Fig.2,3). A reduction in liquid volume below ~ 10% necessitates pressurised sintering (hot die pressing - H.P., or hot isostatic pressing - H.I.P.) in a quest for improved high-temperature properties. An alternative is the post-sintering crystallisation of the glass phase of controlled chemistry; the example given in Fig.3 is that of Nd-Si-Al-O-N glass crystallisation at constant composition [2] and is an alternative to partitioning into oxide/oxynitride combinations used in a commercial Sialon ceramic [3] (Sialon 201 is a  $\beta$ ' $Si_3N_4$  - YAG diphasic ceramic - Fig.2).

Post-sintering heat-treatments are also used in generating the diphasic, cubic + tetragonal, microstructure of a zirconia ceramic which has been 'partially stabilised' by additions of MgO, CaO or  $Y_2O_3$  (PSZ - Fig 3,4). This is heat treated in the 2-phase field to develop tetragonal  $ZrO_2$  particles which are constrained by the matrix and hence retained in metastable form to ambient temperature [4]. The development of a 'critical' mean particle size for the dispersion is such that it is resistant to spontaneous transformation to the monoclinic form but may be transformed in the stress field of a matrix crack, for effective toughening. A similar condition applies to the grain size in a TZP monophase ceramic and in both cases the critical size is a function of stabilising-additive chemistry and concentration.

There have been developments in a range of diphasic ceramics in which various matrices contain dispersions of  $ZrO_2$ , for improved toughness (See Sect.3). These may be viewed as 'composites' with particle dispersions since they are normally non-interactive, artificial, phase combinations produced by mechanical mixing, unlike the diphasic microstructures which result from liquid-phase sintering or solid-state precipitation.

Although the ceramist is now able to induce a variety of microstructure in laboratory-prepared 'billets' by tailoring composition, additive chemistry and sintering/heat-treatment schedule, this is sometimes in conflict with component-shaping capability. Homogeneity is essential for uniform sintering-shrinkage and dimensional precision without the need for final diamond machining. The simpler shaped preforms may be cold die-pressed or iso-pressed in a flexible mould. More complex shapes require injection-moulding with the added problem of 'burn-out' of organic additives.

The problem is compounded if hot-isostatic-pressing is required during the sintering, for example, of high-temperature nitride ceramics with a limited volume of liquid sintering aid. However, impressive dimensional precision has been obtained using glass-encapsulated HIP of injection-moulded preforms at ASEA-cerama in Sweden [5].

## 2.2 Properties

A comparative survey of typical fast-fracture parameters (Modulus of Rupture-MOR and Fracture Toughness- $K_{Ic}$ ) for a range of monolithic ceramic microstructures is presented in Fig 5. A sample of composite-ceramic properties is also included for comparison and discussed in the next section.

The most important characteristic of data for most monolithics is the relatively low 'plateau' level for  $K_{Ic}$  with maximum values of  $\sim 8 \text{ MPa.m}^{1/2}$  for diphasic 'Syalon' ceramics down to 3 or 4  $\text{MPa.m}^{1/2}$  for monophase SiC or  $\text{Si}_3\text{N}_4$  - based microstructures; the differences are mainly associated with increased energy-absorption due to crack-deflection and grain 'pull-out' in diphasic systems with strong morphological anisotropy [6]. Fracture stress (MOR) normally follows the  $K_{Ic}$  trend via the Griffith/Orowan relation  $\sigma = K_{Ic} \cdot C^{-1/2} \cdot \frac{1}{Y}$ , where C is the size of fracture-initiating flaw and Y is a geometrical factor ( $\sim \sqrt{2\pi}$  for semicircular surface flaws). Thus high levels of fracture-stress may only be obtained with refined processing, to eliminate heterogeneities, and with 'low-damage' surface finishing. A simplistic illustration of this relationship (Fig.6) shows that flaw sizes for most monolithics ( $K_{Ic} \sim 5$ ) must be  $\ll 100 \mu\text{m}$  for high strength ( $> 0.5 \text{ GPa}$ ). These are in a size range not easily accessible to NDE techniques and may have a sample distribution which leads to an overlap of strength and service-stress (Fig.6).

Hence, the importance of a substantial increment in  $K_{Ic}$  for  $\text{ZrO}_2$  - based ceramics is clear (Fig.5). It results either from stress-induced transformation of tetragonal  $\text{ZrO}_2$  by the primary crack or its interaction with an array of microcracks induced by particles or grains above a critical size for metastable retention of the tetragonal form. Abnormally high strengths, in excess of 1.5 GPa, have been obtained for TZP ceramics in which modest  $K_{Ic}$  levels (8-10  $\text{MPa.m}^{1/2}$ ) are combined with homogeneity in processing [13], resulting in small flaw sizes with narrow size distribution. Of much greater significance is the ability to obtain high  $K_{Ic}$  values (up to  $\sim 30 \text{ MPa.m}^{1/2}$ ) with some sacrifice to strength [14] (Fig.7). The origin of this effect is not completely understood but is related to additive chemistry and sintering cycle which, in turn, influences the relative contribution of 'transformation' or 'microcrack' toughening. The calculated values of critical flaw size (c), based on the relation shown in Fig.6, are in the mm regime for strengths below 0.5 GPa and  $K_{Ic} \sim 30 \text{ MPa.m}^{1/2}$ . The implication is that such flaws may be generated by microcrack-linkage during sub-critical crack growth and these ceramics may be 'damage-tolerant' and result in a narrowing of the distribution in sample fracture-stress [15]. The high energy-absorption due to microcracking is also manifested as a limited 'pseudo-plastic' stress-strain relation (Fig.7b). The technological influence of this behaviour on engineering design, relaxing some of the statistical constraints of brittle fracture mechanics, is far-reaching. The extension of this phenomenon to a wider range of ceramics, perhaps via development of dispersion-toughened composites is highly desirable.

High-temperature mechanical properties of monolithics are dominated by time-dependent mechanisms such as creep-cavitation. The Si-based ceramics ( $\text{Si}_3\text{N}_4$ , SiC and Sialons) are currently the only contenders for applications above  $\sim 1000^\circ\text{C}$ . The various toughening mechanisms which give  $\text{ZrO}_2$ -based ceramics their exceptional low temperature properties, progressively disappear as the temperature approaches that for the tetragonal-monoclinic transformation and the thermodynamic 'driving-force' is lost (Fig.5). In the contrast the reduction in

strength and  $K_C$  for the Si-based ceramics is small until they reach the softening point of intergranular glassy residues in liquid-sintered compositions. Thus the best strength-retention is for solid-state sintered SiC and then for  $\text{Si}_3\text{N}_4$ -based ceramics in which the liquid is transient (HP Sialon) or is fully crystallised (as in a 201 grade Sialon ceramic). This gradation is replicated in high-temperature creep and creep-rupture (Fig.8,9) although at intermediate temperatures creep-rupture in SiC ceramics occurs at lower stresses than for  $\text{Si}_3\text{N}_4$ , reflecting their failure mode from pre-existing flaws. When failure occurs by an accumulation of creep-cavitation damage the gradient of the stress-rupture plot is relatively high, exemplified by typical commercial  $\text{Si}_3\text{N}_4$  ceramics with glass-residues at grain junctions (Fig.9).

A temperature ceiling for  $\text{Si}_3\text{N}_4$ -based ceramics is frequently dictated by oxidation-induced degradation of microstructure, which is catalysed by a reaction between the intergranular phase and an initially-protective surface  $\text{SiO}_2$  film [16]. Hence, for the highest temperatures, monophasic SiC ceramics are favoured. There is a need to further develop HIPped  $\text{Si}_3\text{N}_4$  ceramics with minimal residual phases or those which are stable in relation to  $\text{SiO}_2$ . Such microstructures should also be more resistant to degradation under hot-corrosive conditions.

### 3. STRUCTURAL COMPOSITES

There are two broad classes of ceramic-matrix composite in which there has been a rapid escalation of research interest (Fig.10). The main motivation for this is the possibility of removing the severe engineering design constraint imposed by brittle fracture mechanics.

The first class of composite is that in which a random dispersion of particles or whiskers is contained in a ceramic matrix with the objective of increasing its fracture toughness. The dispersed phase normally occupies <30 vol.% such that conventional powder mixing and sintering may be used in fabrication although pressure-assisted sintering is required for whisker dispersants. Toughening mechanisms are varied and may operate simultaneously, dependent on dispersed phase morphology, thermal expansion mismatch and interface type. They include crack-deflection [17], bridging and pull-out or interface microcracking [18] in the crack-tip stress field (Fig.11). The latter mechanism is initiated by thermal mismatch or, in the special case of  $\text{ZrO}_2$  dispersants, by phase transformation. It requires a critical particle size below that for general microcracking and its origin dictates only a low-temperature effectiveness. Apart from TZP, particle-toughening of various matrices ( $\text{Al}_2\text{O}_3$ , mullite, cordierite,  $\text{Si}_3\text{N}_4$ ) useful increments in  $K_C$  have been obtained for dispersions of  $\beta$  SiC whiskers in  $\text{Al}_2\text{O}_3$  [10], TZP [19], glass-ceramics and  $\text{Si}_3\text{N}_4$  [11] (Figs.5 and 12). There is some encouraging data for transition metal borides or carbides (e.g.  $\text{TiB}_2$ ,  $\text{TiC}$ ) in matrices such as SiC [12] and  $\text{Si}_3\text{N}_4$  and there is a need to develop a greater range of dispersed-phases in anisotropic morphology and controlled size. The levels of  $K_C$  attainable via. dispersions are of order 2-3x the matrix value and hence rarely exceed 15  $\text{MPa m}^{1/2}$ . However, this is significant in relation to its influence on critical flaw size or the shift and narrowing of fracture-stress distribution away from that encountered in service (Fig.6).

A dispersion-containing composite is not always fabricated with the object of increasing  $K_C$ . At a critical volume fraction of dispersed phase (~30%) the particles may form an interconnected network such that additions of transition-metal nitrides or carbides confer electrical conductivity on the otherwise insulating ceramic. In this state it may be electro-discharge machined (EDM) into high-precision engineering components; the example in Fig.13 is a commercial Sialon 501 ceramic in which TiN is the dispersed phase [20].

A second class of composite is based on the totally different concept of load-transfer to high modulus semicontinuous fibres during matrix-microcracking in a service overload situation (Fig.14). This mechanism has been demonstrated for unidirectional composites in tension both via. the stress-strain relation and observation of matrix-microcracking [21]. The important implication is that, apart from high failure strain and tolerance of transient overloads, the ceramics become insensitive to matrix flaws and simple fracture mechanics principles (Fig.6) are no longer applicable [22]. The achievement of this optimal stress-strain response depends not only on axiality of loading but also on a high volume fraction of fibres with a relatively-weak interface bond. The latter is function of fibre/matrix chemistry and processing parameters; reactive interfaces promote crack-continuity and a reversion to monolithic behaviour. Availability of suitable fibres is a major limitation, developments so far having concentrated on carbon and SiC produced from yarn precursors. 'Nicalon' and 'Tyrrano' are commercial SiC fibres which are non-crystalline and contain high oxygen impurity levels. These non-stoichiometric 'oxycarbides' undergo a fortuitous interdiffusive reaction with refractory glasses (such as the cordierite [MAS] or spodumene [LAS] glass-ceramic precursors) to yield carbon-rich interfaces [23] which have low cohesion (Fig.15). Thus a number of Nicalon-glass ceramic composites have been developed, based on earlier research at United Technologies Research Labs. (U.S.A.) [24]. An alternative is to use low-temperature infiltration of matrix phases via. the vapour phase (CVI-Fig.10), to give composites such as SiC-SiC, C-C and SiC-C [25]. This infiltration process eases the problem of 3-D composite fabrication via. the ability to weave shaped preforms prior to infiltration. However, in cross-ply form the effective values of  $K_C$  show little improvement on monolithics and the infiltration problem results in residual porosity.

~~Apart from the increased difficulty in fabrication and net-shaping of composites~~ there are many remaining problems associated with their application, especially at high temperatures and in oxidising conditions [24]. Degradation of the critical C-rich interface occurs in oxidising atmospheres for Nicalon-type fibres (Fig.15). The oxidative degradation of C-C or SiC-C composites above 800°C has been alleviated by SiC coating, extending their potential service temperature to >1400°C [26]. However, the yarn-based amorphous SiC fibres have microstructural instabilities above ~1200°C, with associated strength-loss. Hence there is a need for high-stability stoichiometric SiC fibres of 'weaveable' diameter (10-20µm) which also have negligible matrix reactivity during high-temperature fabrication, thus introducing the possibility of refractory matrices (e.g. Si<sub>3</sub>N<sub>4</sub>) via. conventional pressure-assisted sintering. Such fibres have recently been produced by MERC, Arizona (USA) and may be available commercially within 1988. A decrement in fabrication temperature for Si<sub>3</sub>N<sub>4</sub> matrices is obtained via. the reaction-sintering route (RBSN) in which Si powder is nitrified at 1400°C 'insitu', as a matrix for SiC fibres. The concept of load transfer following matrix microcracking has recently been demonstrated for these composites, with ultimate fracture stresses up to 1GPa [27].

A particularly innovative processing route for composites is the 'Lanxide' process (Lanxide corp., U.S.A.[28]). It is applicable to oxide, nitride, boride or carbide matrices with particulates or continuous fibres. For example SiC fibre-reinforced composites have been fabricated with Al<sub>2</sub>O<sub>3</sub> matrices by in-situ directional oxidation of liquid Al. The claimed advantages are a negligible-shrinkage and near-net shaping of large components at low projected cost. Typical ambient temperature properties quoted for the SiC-Al<sub>2</sub>O<sub>3</sub> matrix composite are a strength (MOR) of nearly 800 MPa coupled with fracture toughness of 27 MPa.m<sup>1/2</sup>. Part of this high  $K_C$  value may arise from the constrained plasticity of a residual interconnected metallic phase (5-30%) such that it may impose a limitation to high-temperature applications.

#### 4. APPLICATIONS

The dominant principle for application of ceramics in the engineering and process industries is a balance between the identity of property superiority over metallic or polymeric systems and the more difficult fabrication, increased cost or low 'reliability' of ceramic components. Hence they have been contenders for applications in the low-temperature regime which require high hardness, wear and corrosion resistance or low friction. These potential benefits may remain at elevated temperatures and are supplemented by creep, oxidation/corrosion resistance and dimensional stability.

Established applications are mainly for monolithic ceramics and inevitably lie in the relatively 'low-risk' areas such as metal cutting/abrasion/forming/melting and as seals, bearings or impellers working under harsh conditions. For the low-temperature applications  $\text{Si}_3\text{N}_4$ -based ceramics and  $\text{ZrO}_2$ -based ceramics are competitors; examples of the tool tips, drawing/extrusion dies, valve and pump components are illustrated in Fig.16. There are established benefits in relation to tool or component lifetime, cutting/forming speed (Fig.16) or surface finish and dimensional precision of the metallic work-piece. SiC whisker-toughening has given  $\text{Al}_2\text{O}_3$ -based ceramics a new potential; tool tips are now commercially viable due to the significant increment in fracture-toughness and thermal shock resistance. It is probable that future ultra-high precision, 'nanotechnology', finish-machining of metals and ceramics will require machine-tool beds and bearings constructed from ceramics, for high mechanical and thermal stability.

The 'high-risk' areas are those involving much greater component cost, increased stress, temperature and hence failure probability, exemplified by the various energy-conversion systems with the object of better fuel-efficiency or performance. Most ceramic applications remain at a development stage after more than 10 years of ceramic substitution in both turbines and reciprocating engines. Rare examples of commercial components are the turbocharger rotor ( $\text{Si}_3\text{N}_4$ -based Nissan/NTK), valve rocker-arm pads ( $\text{ZrO}_2$  and  $\text{Si}_3\text{N}_4$ -Kyocera/Isuzu, NTK/Nissan, Bosch). Various components, aimed at insulation/higher-temperatures and hence thermal efficiency, are at or near-commercial production, for example, piston crown, cylinder and exhaust port liner (Porsche, Audi, Kyocera/Isuzu, - either  $\text{Si}_3\text{N}_4$ ,  $\text{ZrO}_2$  or aluminium titanate). However, the general conclusion for insulated systems is that to gain the large predicted increments (up to ~30%) in efficiency the energy in the much-hotter exhaust gases must be recovered via. a turbo-compound engine configuration. There has recently been an important DTI initiative in this area (the CARE programme) involving over 20 UK companies, from engine manufacturers to ceramic developers. There have been longer-term developments in turbine-component substitution in the UK, Germany, Sweden and, particularly, in the USA via. the large Department of Energy-sponsored programmes. Hot gas-flow components (blades, stators, shroud-ring) have been mainly substituted by  $\text{Si}_3\text{N}_4$  or SiC ceramics, combustors and heat exchangers mainly by SiC and silicate glass-ceramics, respectively. Some examples of engine components (most of which are diamond machined from ceramic billets in the development stage) are given in Fig.17.

The major factor inhibiting commercial uptake in these high risk areas, apart from economical net-shaping, is the reliability/fracture toughness problem for monolithic ceramics. There are currently some encouraging composite developments which are near commercial acceptance although not at the high stresses, temperatures and long lifetimes projected for turbines. For example, CVI SiC/SiC fibre - wound heat exchanger tubes (Babcock & Wilcox Co. U.S.A.) and woven Nicalon or carbon fibres with CVI SiC for aerospace applications (as re-entry thermal protection or ramjet combustor liners - Societe' Europeene de Propulsion, France).

## 5. FUTURE PROSPECTS AND BONDING PROBLEMS

There is already a sophisticated level of microstructural design and property optimisation in monolithic ceramics, exemplified by the use of specific additive chemistries and post-sintering transformations in  $\text{Si}_3\text{N}_4$  and  $\text{ZrO}_2$  based ceramics. There is a need to couple the enhanced fracture-toughness levels attainable in  $\text{ZrO}_2$  ceramics with the high-temperature potential of  $\text{Si}_3\text{N}_4$  and  $\text{SiC}$  ceramics. This will not come from further microstructural refinements of existing monolithic ceramics but there is a promise of modest increments in  $K_{IC}$  via dispersion toughening and its application to a wider range of matrices, including the oxides. Hence there is a requirement for commercial availability of dispersants in platelet or whisker form, covering a range of chemistry.

The greatest advances, into the 1990s, will be made in ceramic-matrix composites containing semicontinuous fibres. For low-temperature fabrication, CVI of 3-dimensional woven preforms will require greater efficiency and reduced cost if these composites are to be accepted for diverse engineering applications. Glasses and glass-ceramics are alternative low-temperature matrices, enhanced by sol-gel preparative routes, with a possibility of woven preforms from glass-coated fibres. For high-temperature application there is a need for weavable high-stability fibres such as stoichiometric  $\text{SiC}$ , preferably from yarn precursors. Fibre coatings may be required for non-reactive interfaces during fabrication and in extended use under oxidising conditions.

Ceramic/ceramic or ceramic/metallic joining with high-levels of cohesion normally requires an interdiffusion or chemical reaction at the interface, using methods described elsewhere in this conference proceedings (e.g. in the paper by Nicholas). Hence the simplest ceramic materials for joining normally have monolithic and monophase microstructures. An example is the active metal brazing of a hot-pressed  $\text{Si}_3\text{N}_4$  ceramic in which the origin of cohesion is a microlayer of  $\text{TiN}$  formed via reaction of the ceramic surface and  $\text{Ti}$ , which is an additive to a conveniently-melting alloy, such as a  $\text{Cu-Ag}$  eutectic. Even this simple system may be complicated by the formation of additional interfacial compounds, such as metallic silicides (Fig. 18). The situation is further exacerbated in multiphase monolithic ceramics (such as pressureless-sintered sialons) and especially in composites. The latter materials are essentially heterogeneous with extreme diversity in phase-chemistry, on a scale normally in the range 10-100  $\mu\text{m}$ , coarser than in multiphase monolithic ceramics. Hence ceramic-matrix composites will present an additional challenge in bonding technology, with problems which may be anticipated thus:-

- (i) There will be multiple interdiffusion or reaction paths between substrate or braze-layer and the composite which may inhibit the formation of a simple cohesion-layer (Fig. 19).
- (ii) In structural ceramics higher levels of bond-strength will be required to match the increased failure stresses in composites. Part of this problem is the discontinuity of microstructure in the interface which destroys the composite property of flaw insensitivity or fracture-toughness.
- (iii) In composites requiring oxidative or corrosion protection, surface coatings may be influenced by the bonding reaction (Fig. 19).

For structural ceramics generally, there will be a requirement for joining of component surfaces having complex shapes. All these factors present formidable problems to the science and technology of ceramic joining into the next century.

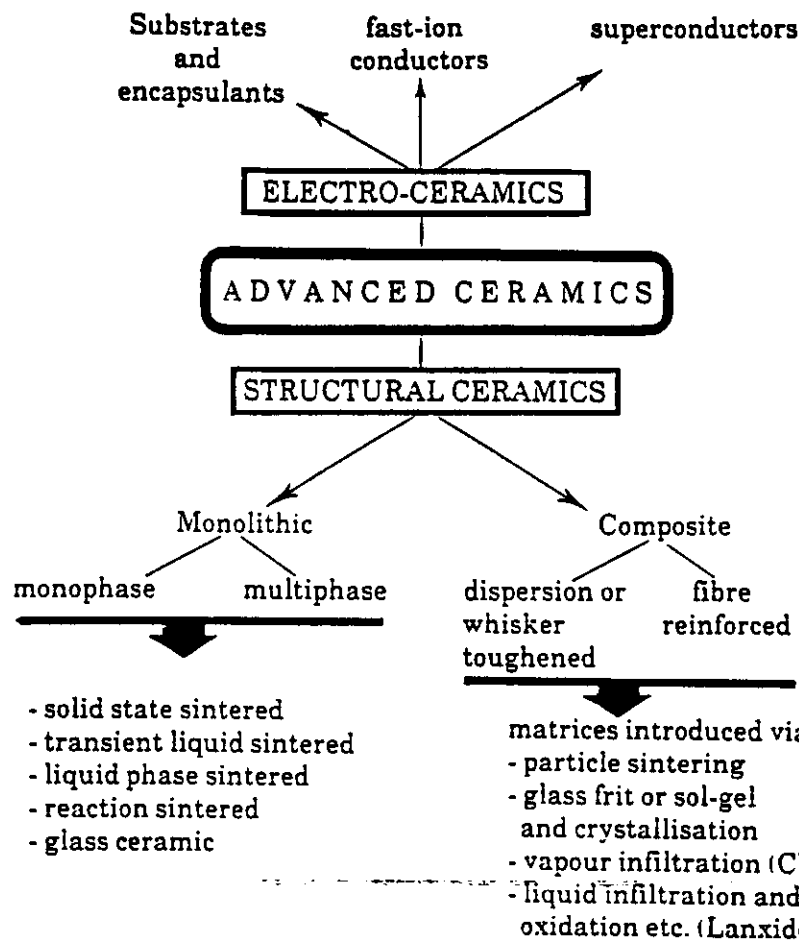


## ACKNOWLEDGMENTS

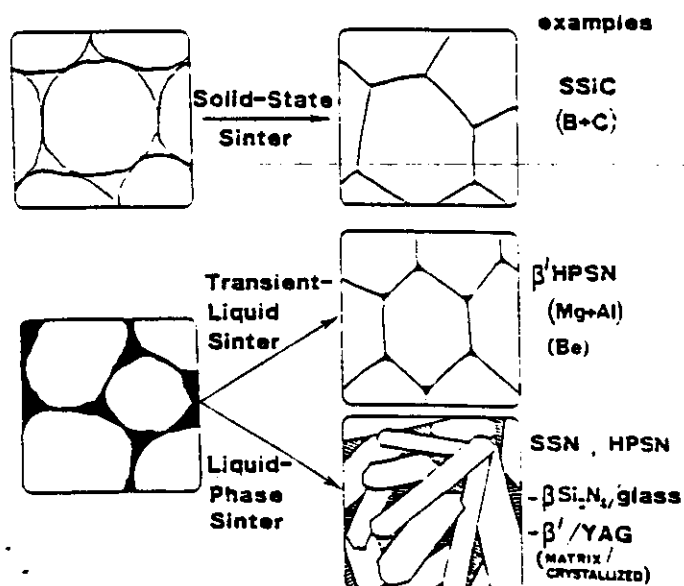
The author wishes to acknowledge the sponsorship of a number of ceramics research programmes at Warwick by Rolls-Royce plc, Lucas-Cookson-Syalon Ltd., B.P. Research, GEC, T & N Technology and the SERC.

## REFERENCES

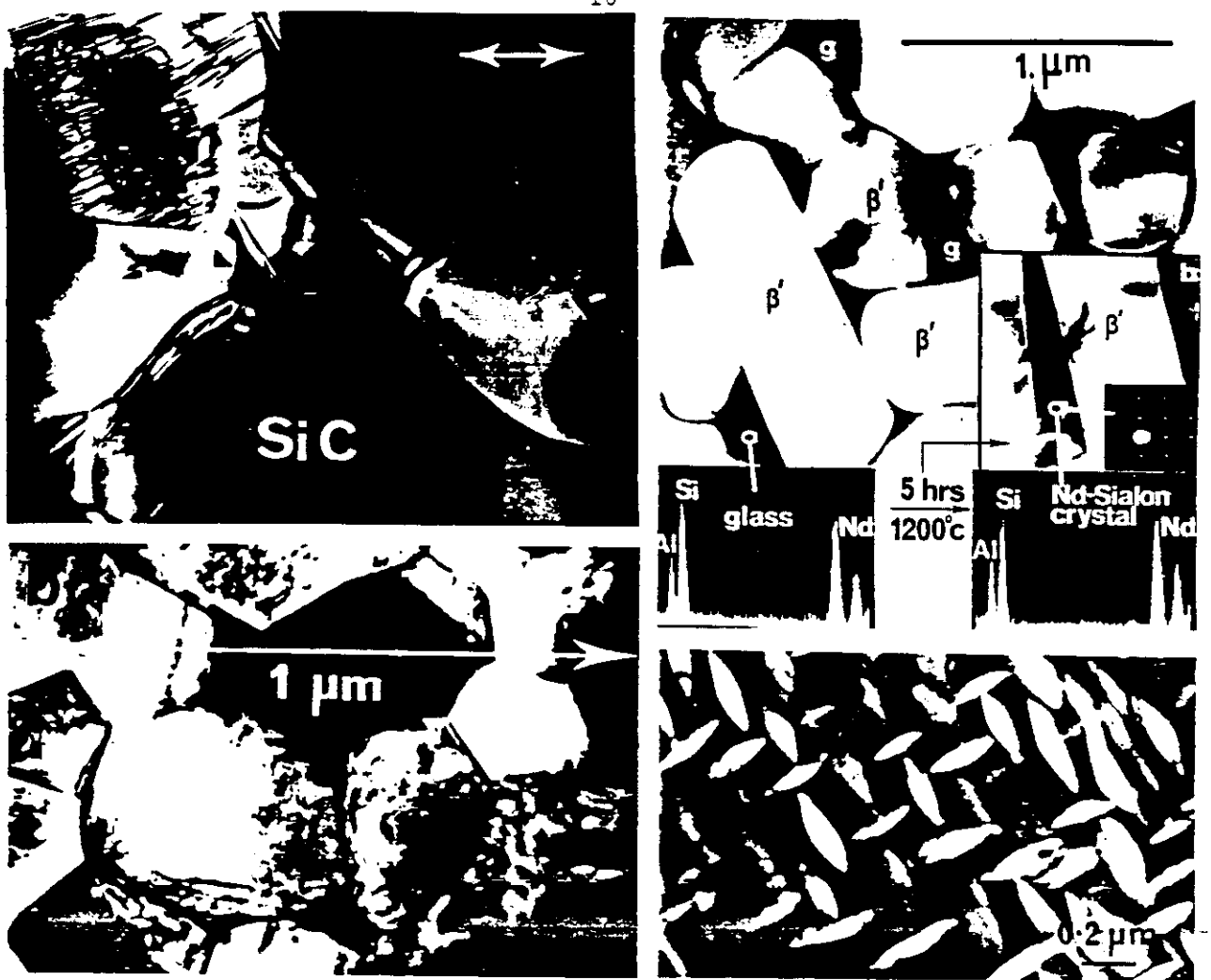
1. Lewis MH and Lumby RJ: Powder Metall. 1983 26 73
2. Lewis MH, Leng-Ward G and Jasper C: in proc. 1st Int. Conf. on Ceramic Powder Processing Science 1987, Amer.Ceram.Soc. (in press).
3. Lewis MH, Bhatti AR, Lumby RJ and North B: J.Mat.Sci 1980 15 103
4. Stevens R: "Zirconia and Zirconia Ceramics" 1986 (Magnesium Electron publication No 113)
5. Larker HT: Mat.Sci.Res. 1984 17 571
6. Lewis MH, in "Tailoring Multiphase and Composite Ceramics" eds. Tressler, Messing, Patano and Newnham, (Plenum 1986) 713
7. Brennan JJ and Prewo KM: J.Mat.Sci. 1982 17 2371
8. Caputo AJ, Stinton DP, Lowden RA and Besmann TM: Am.Ceram.Soc.Bull. 1987 66 368
9. Gadkaree KP and Chyung K: Am.Ceram.Soc.Bull. 1986 65 170
10. Wei GC and Becher PF: Am.Ceram.Soc.Bull. 1985 64 298
11. Hayami R, Ueno K, Kondo I, Tamari N and Toibana Y: in "Tailoring Multiphase and Composite Ceramics" eds. Tressler, Messing, Patano and Newnham (Plenum 1986) 663
12. Janney MA: Am.Ceram.Soc.Bull. 1987 66 322
13. Claussen N: Mat.Sci. and Eng. (1985) 71 23
14. Tsukuma K, Ueda K and Shiomi M: 38th Pacific Coast Meeting of the Am.Ceram.Soc., Irvine, California, Oct. 1985.
15. Matsui M, Soma T and Oda I: J. Am.Ceram.Soc. 1986 69 198
16. Lewis MH, Mason S and Szveda A: in 'Non-oxide Technical and Engineering Ceramics' ed. Hampshire 1986 (Elsevier) 175
17. Faber KT and Evans AG: J.Am.Ceram.Soc. 1984 67 225
18. Faber KT and Evans AG: Acta.Metall. 1983 34 565
19. Claussen N, Weisskopf KL and Ruhle M: J.Am.Ceram.Soc. 1986 69 288
20. Cain MG: student project report, University of Warwick 1986
21. Marshall DB and Evans AG: J.Am.Ceram.Soc. 1985 68 225
22. Evans AG: Mat.Sci. and Eng. 1985 71 3
23. Brennan JJ: in "Tailoring Multiphase and Composite Ceramics" eds.Tessler, Messing, Patano and Newnham (Plenum 1986) 549
24. Prewo KM: in "Tailoring Multiphase and Composite Ceramics" eds.Tressler, Messing, Patano and Newnham (Plenum 1986) 529
25. Naslain R and Langlais F: in 'Tailoring Multiphase and Composite Ceramics' eds.Tressler, Messing, Patano and Newnham (Plenum 1986) 145
26. Jamet JF: in 'New Materials and their Applications' 1988 Inst.of Physics Conf.Series (Adam Hilger - in press).
27. Corbin ND, Rosetti GA and Hartline SD: Ceram.Eng.Sci.Proc. 1986 7 958
28. Andersson CA and Aghajanian MK, 12th Annual Conf.on Composites and Advanced Ceramics, Am.Ceram.Soc., Cocoa Beach 1988
29. Ramsey MJ and Lewis MH: Mat.Sci. and Eng. 1985 71 113



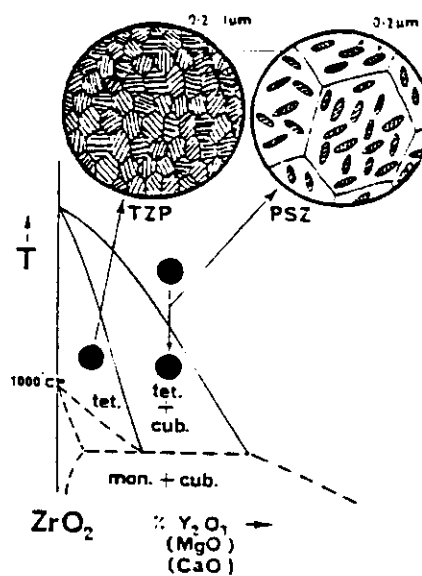
## 1 Classification of advanced ceramics



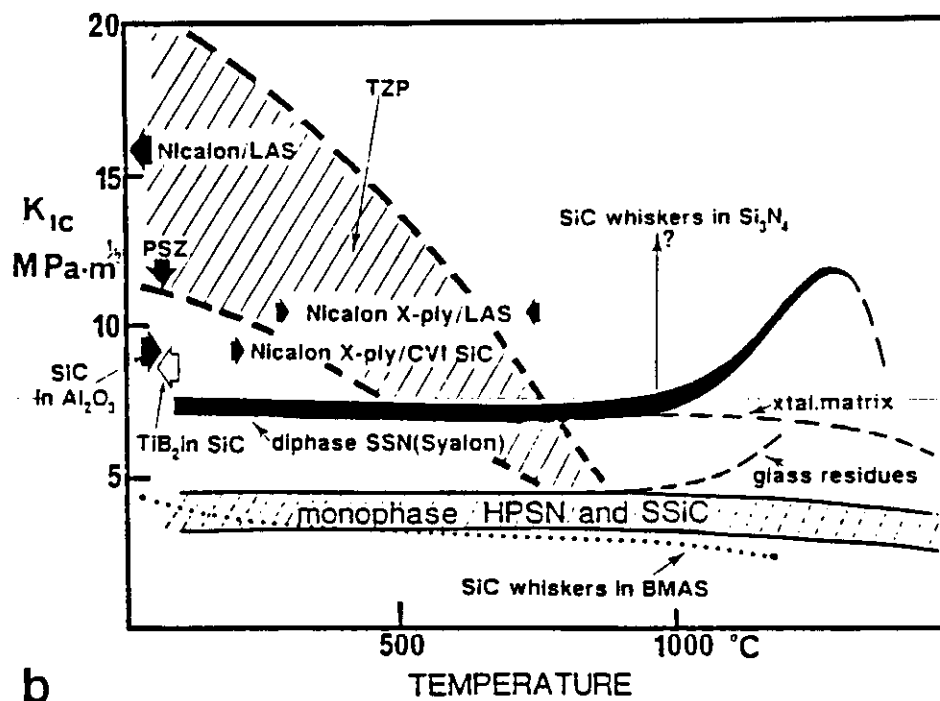
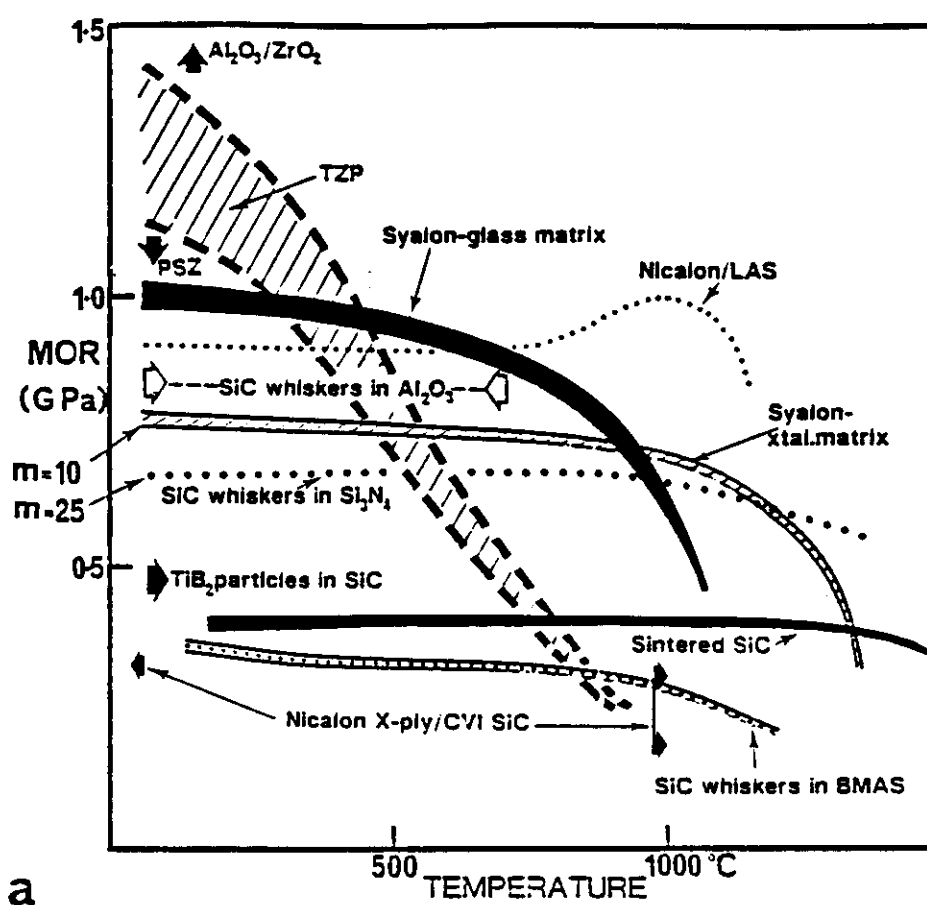
## 2 Microstructural types and processing routes for monolithic ceramics



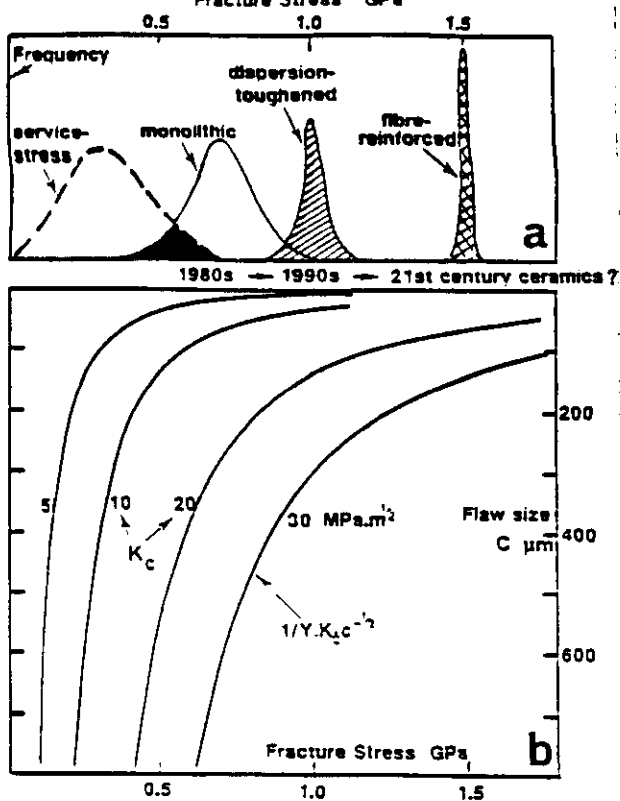
- 3 Monolithic ceramic microstructures; (a) Hot-pressed SiC (b) Sintered tetragonal ZrO<sub>2</sub> (TZP) (c) Sialon sintered with Nd<sub>2</sub>O<sub>3</sub> additive and matrix-crystallised (Ref.2) (d) Partially-stabilised ZrO<sub>2</sub> (PSZ with MgO-addition-courtesy Prof. A.H. Heuer)



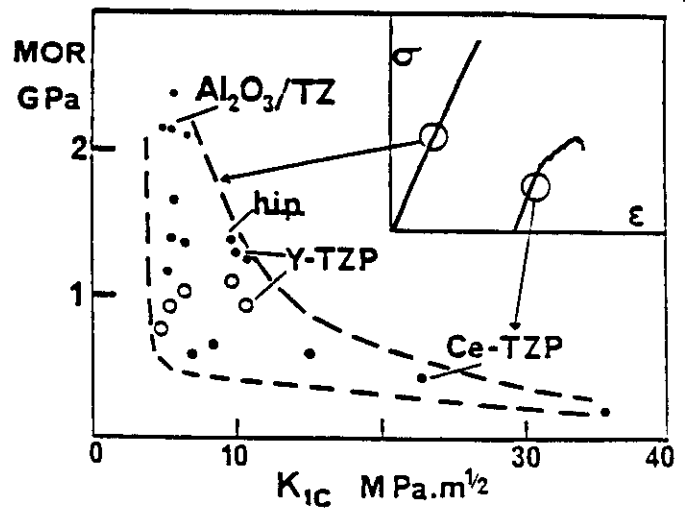
- 4 The relation between phase-equilibria and microstructure for TZP and PSZ ceramics.



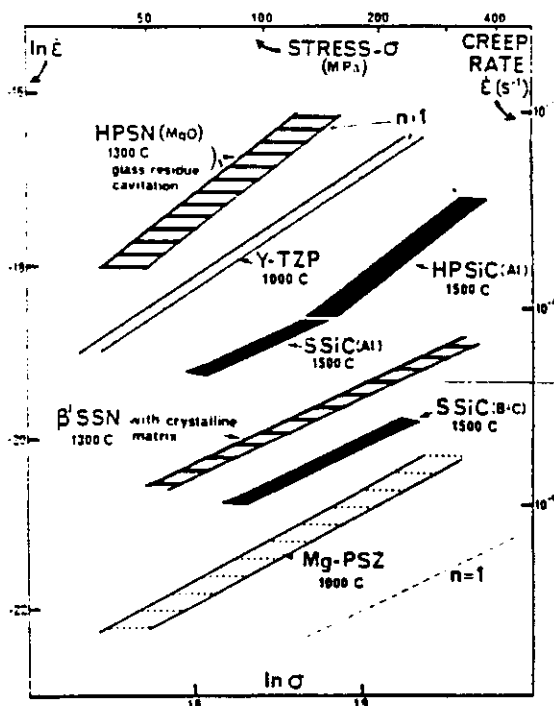
- 5 A comparison of, (a) strength (modulus of rupture-MOR) and (b) fracture-toughness ( $K_{Ic}$ ) for a range of monolithic and composite ceramics, with varying temperature. Nicalon in LAS [7], Nicalon/CVI SiC [8], SiC in BMAS [9], SiC in  $Al_2O_3$  [10], SiC in  $Si_3N_4$  [11],  $TiB_2$  in SiC [12],  $ZrO_2$  ceramics [4], Sialon ceramics [6].



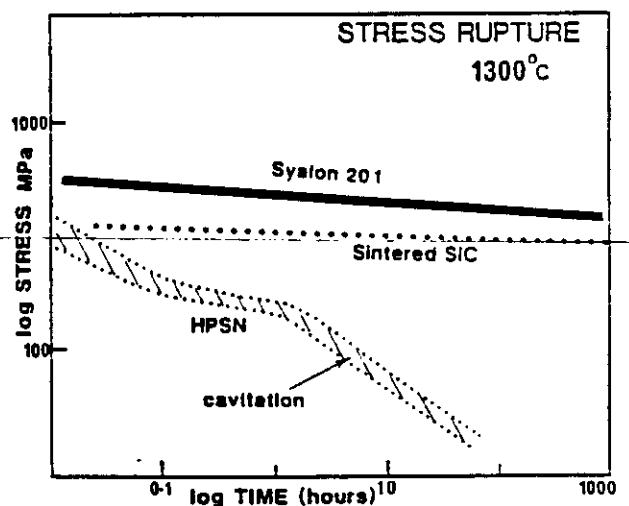
6 The relation between fracture-stress, fracture-toughness and flaw size in brittle fracture-mechanics. In (a) an idealised shift in sample strength-distribution with composite development is illustrated.



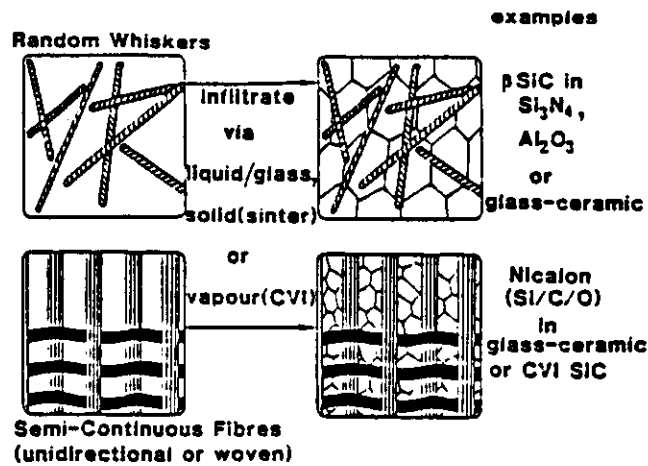
7 The variation in fracture stress and fracture-toughness with microstructure and additive in  $ZrO_2$ -based ceramics (after Ref.14). High  $K_{IC}$  ceramics may exhibit microcracking-induced pseudo-plasticity (inset).



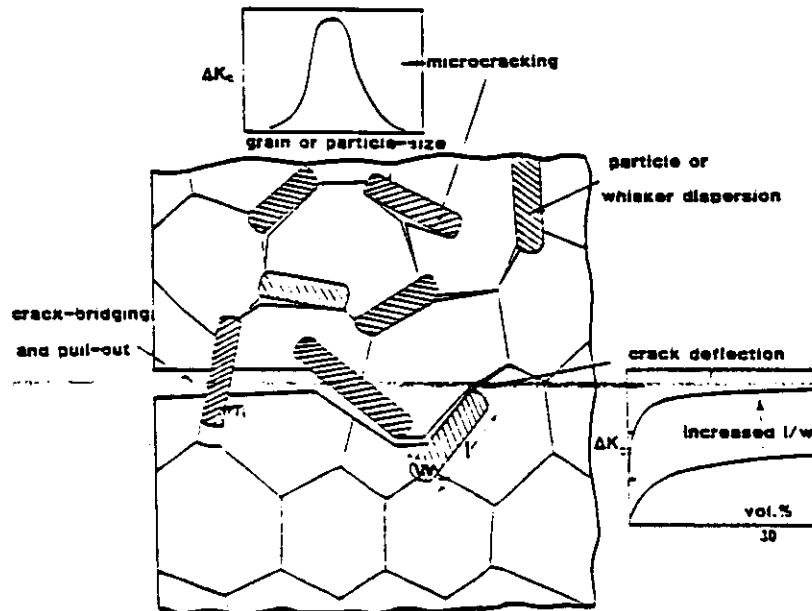
8 A comparison of pseudo-steady-state creep rates for a range of Si-based and  $ZrO_2$ -toughened ceramics in the 1000°C-1500°C interval.



9 Stress-rupture comparison for Si-based ceramics illustrating the detrimental influence of creep-cavitation.



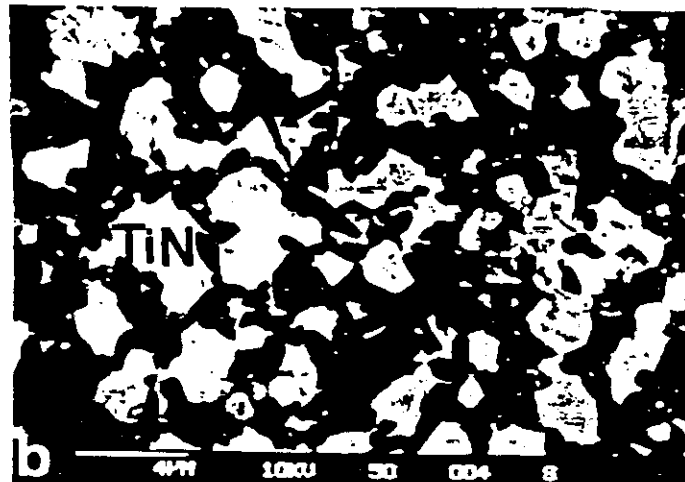
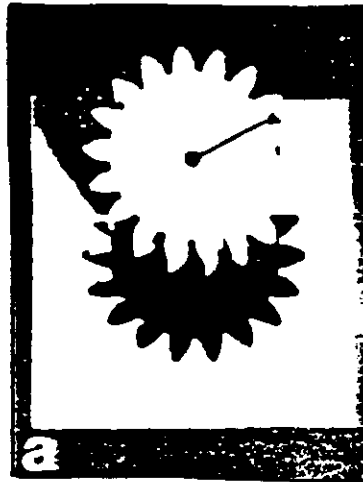
10 Microstructural types and processing routes for composite ceramics.



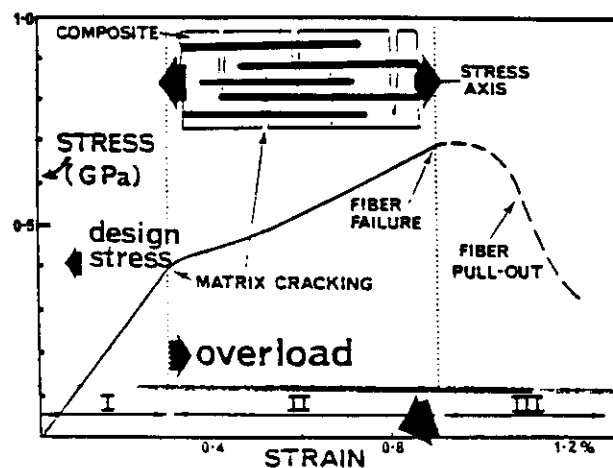
11 Mechanisms contributing to enhanced fracture-toughness ( $K_c$ ) in dispersion-toughened composites.



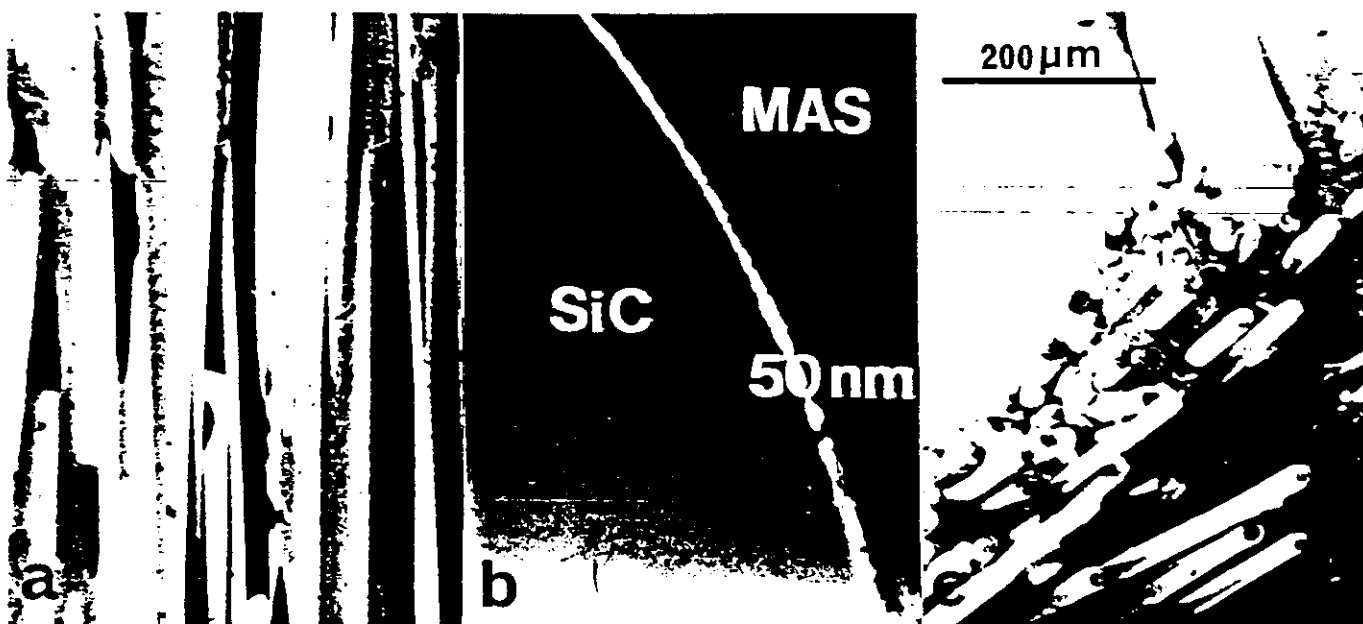
12 Examples of whisker and particle-toughened microstructures: (a) tet- $\text{ZrO}_2$  in  $\text{Al}_2\text{O}_3$  - (courtesy Prof. D.J. Green), (b) SiC whiskers in glass-ceramic with fracture-surface in (c) (unpublished work Li Jie, Univ. of Warwick).



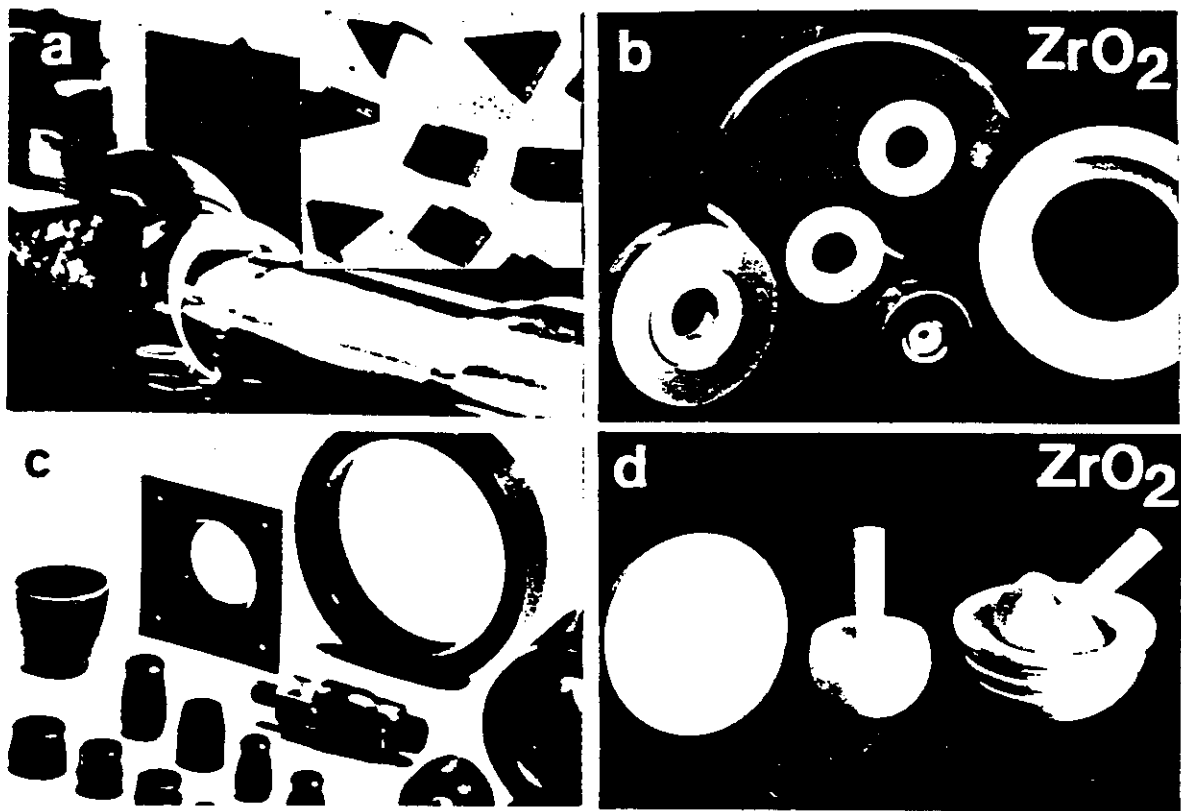
- 13 An electro-discharge-machinable ceramic (Syalon 501); (a) precision-cut component, (b) TiN - Syalon microstructure. (Ref.20).



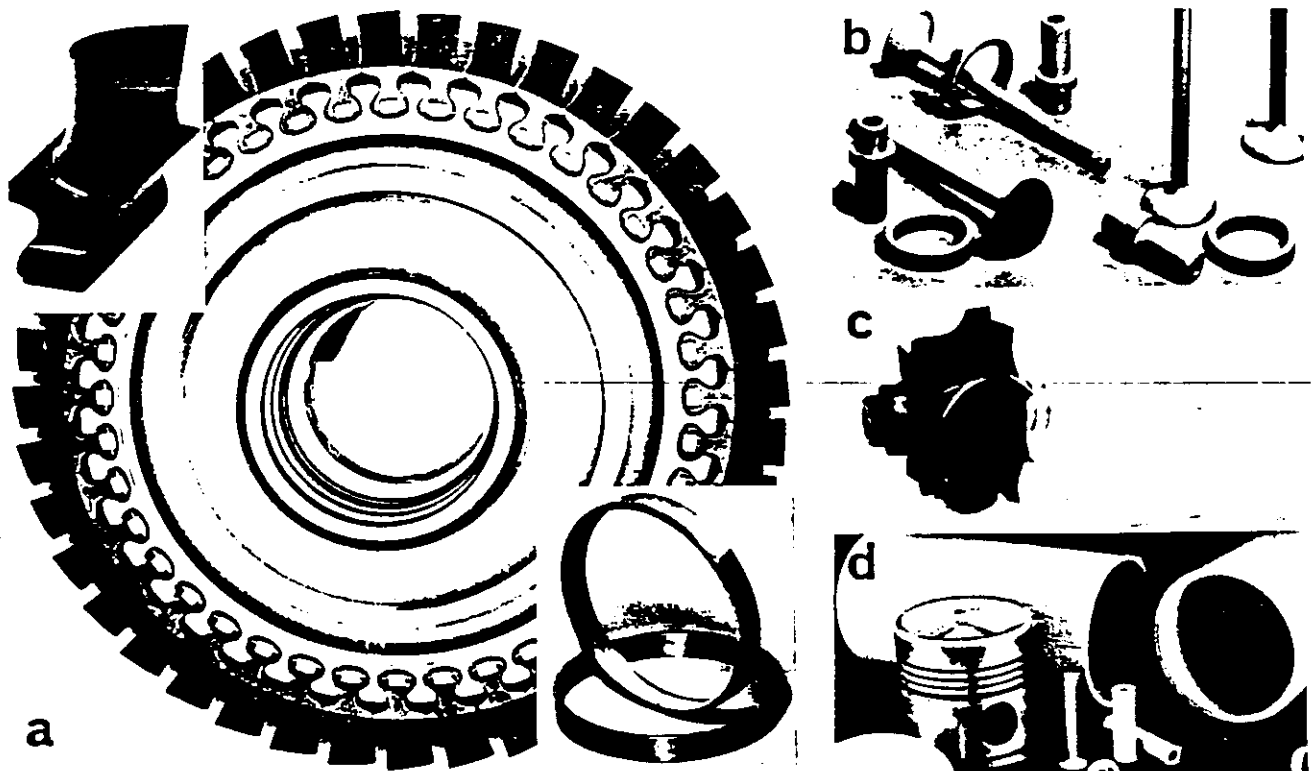
- 14 The optimal stress-strain behaviour of a fibre-reinforced composite, associated with matrix microcracking and load-transfer beyond the maximum design stress.



- 15 Microstructures of SiC (Nicalon) reinforced glass-ceramic (MAS-Cordierite) illustrating the C-rich low-cohesion interface-(b) and fibre pull-out in fracture-(c). (courtesy Dr. VSR Murty - Univ. of Warwick).

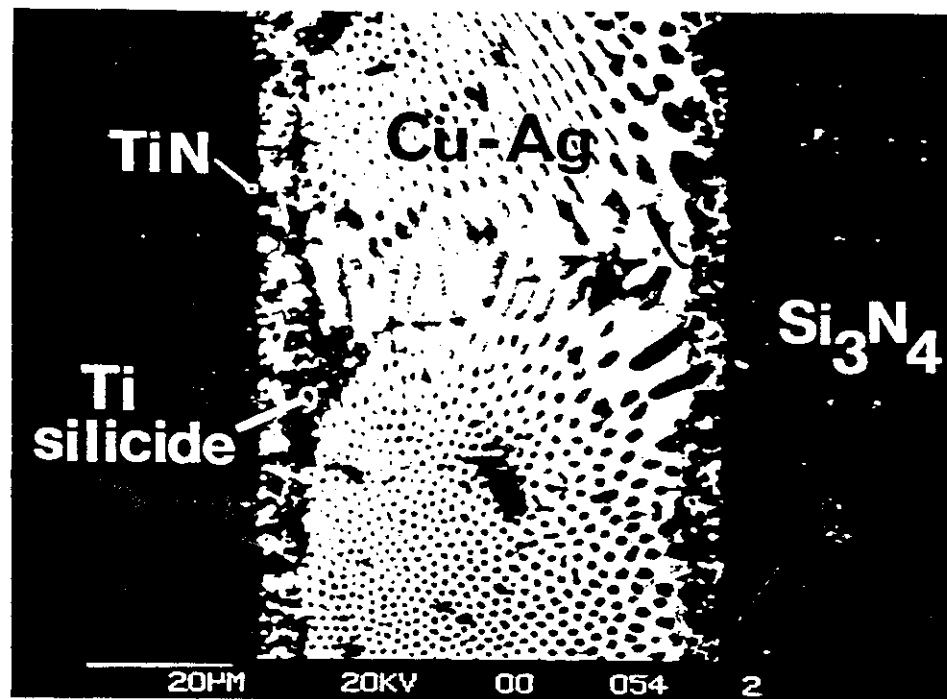


- 16 Monolithic ceramic components ( $ZrO_2$ -based or Si-based) for tool-tips-(a), metal forming dies-(b) seals, bearings, crucible (c) and medical prosthesis-(d). Syalon components-courtesy of Lucas-Cookson-Syalon Ltd,  $ZrO_2$  - courtesy Nilcra Ceramics Pty Ltd.

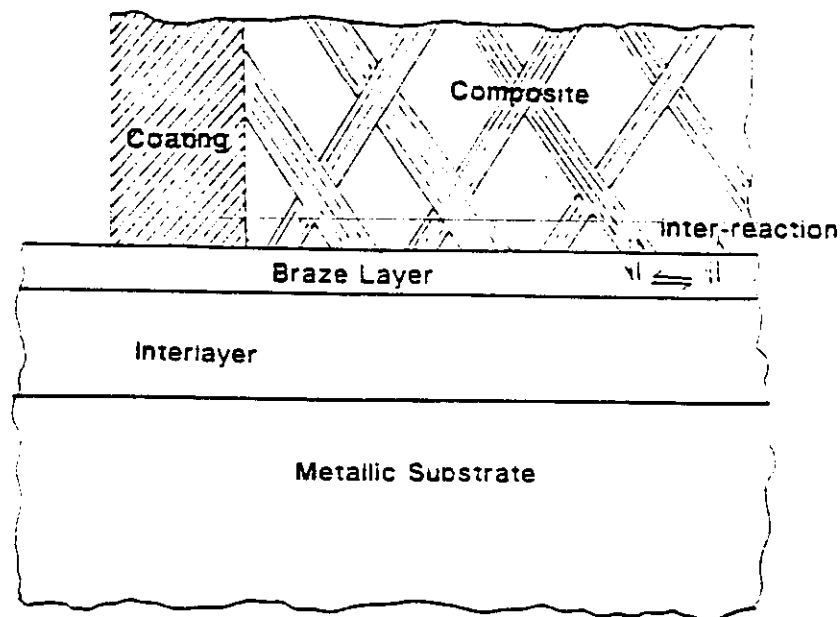


- 17  $Si_3N_4$ -based components for heat-engines: (a) turbine blades and shroud-ring (Rolls Royce plc) (b) valve components, Lucas-Cookson-Syalon), (c) turbocharger-rotor (NGK Spark Plug Co. Ltd.,). (d)  $ZrO_2$  cylinder liners and piston cap (Nilcra Ceramics Pty).





- 18 Section through an active-metal brazed ceramic/ceramic joint showing the TiN reaction-layer, silicide precipitation and the Cu-Ag eutectic micro-structure [Ref. 29].



- 19 Sectional diagram of a possible bonded interface illustrating the complexity of interaction for composites.

