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ESIS Technical Committee 6 "Ceramics"

Newsletter 32

Minutes of the 20th meeting
8th and 9th of May, 2003
Institut für Struktur- und Funktionskeramik, Montanuniveristät Leoben, Austria

The 20th meeting in Leoben was hosted by Prof. Dr. Robert Danzer of the Institut für Struktur- und Funktionskeramik, Montanuniversität Leoben. 20 participants from 8 countries attended the scientific sessions.

After a welcome by the Rector of the Montanuniversität, Magnifizenz Wolfgang Pöhl, the meeting was opened by Prof. Danzer.

RMTP - ESIS TC6 Reference Material Testing Program

A progress report was given by the program co-ordinator, the topic co-ordinators and the participants. TopicA: Measurements of the thermo-mechnical properties are in progress. Topic B: Some tests have to be completed, some specimens need final machnig. Topic C: Special Tests (tensile, bi-axial, Brazilian disc) are still missing. Fracture toughness measurement are nearly completed. Topic D: The SCCG-Testing Program is finished with the exception a few tests under corrosive environment. In the cyclic fatigue part the R = -1 tests are in progress. Topic E: A new topic coordinator (Jan Dusza) has been appointed. A frist version of the Hat- test program was established, bend specimens were distributed. First results from J. Dusza and H. Klemm indicate, that the test program has to be reviewed!

There are now five publications on the results of the RMTP published, three are submitted or in press.

Scientific Program

Eight scientific presentations were made around the theme "Reliability and Lifetime Prediction of Structural and Functional Ceramics". Abstracts of these presentations are part of these minutes.

Election of new chairpersons

After approx. ten years of taking care of ESIS TC6 Prof. Dr. Robert Danzer wanted to hand over the responsibility of chairing this committee into new hands. A call for candidates for the positions of a chair and a vice-chair yielded two nominations. The election was held as a combination of an election by mail and directly at the meeting. The results are as follows:

Chair	**Vice-Chair**
Assoc.Prof Dr. Jan Dusza, IMR-SAS Košice, Slovakia:	**Dr. Tanja Lube**, ISFK, Montanuniversität Leoben, Austria:
55 ballots distributed, 32 returned	55 ballots distributed, 32 returned
31 accepted, 1 invalid	31 accepted, 1 rejected

Both new chairpersons agreed to take over the functions and express their thanks for the support.

Next Meetings etc.

The next (**21th**) ordinary meeting will be held on **March 3rd - 4th, 2004** at the DLR in Köln.

For those members of TC6 who have for some reason not received the recent e-mail by Professor Giuseppe Ferro, the ESIS Secretary, we attach the documents he send concerning the ESIS membership for 2003.

Yours sincerely,

Assoc. Prof. Dr. Jan Dusza Dr. Tanja Lube

Increasing Resistance to Low Temperature Ageing Degradation of Y-TZP by Surface Modification

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It is well known that tetragonal zirconia policrystals stabilised with yttria (Y-TZP) are ceramic materials that exhibit an interesting combination of strength and fracture toughness. The reason for this is their very small grain size and the phase transformation of the tetragonal structure into monoclinic symmetry that is accompanied by a volume expansion. When this is activated by the stress field in front of the tip of a crack, the increase in fracture toughness that results by transformation toughening is proportional to the extent of the transformation, which depends on temperature, the amount and type of stabiliser, and the grain size of tetragonal zirconia.

Unfortunately, this transformation may also take place spontaneously at the surface with the presence of water vapour in the air during ageing at low temperatures (from about 100 to 400 ºC) and the phenomenon is referred to as low temperature ageing degradation of Y-TZP. The volume expansion that accompanies the transformation can generate microcracks in the transformed surface, thereby degrading severely the strength and surface properties of the material.

In the present work, it has been investigated the resistance to low temperature ageing of 2.5% molar Y-TZP with different microstructures: a) a small tetragonal grain size; b) a duplex (tetragonal+cubic) coarse microstructure induced by heat treatment in air at 1650 ºC; c) a duplex (tetragonal+cubic) coarse microstructure with a thin surface layer of alumina produced by a high temperature reaction with AlN powder in a nitrogen atmosphere. The microstructure and mechanical properties that result from these heat treatments have been evaluated. Special attention has been given to the strength, which has been measured in a miniaturised three-on–ball flexure-testing device, and to the fracture toughness, which has been estimated by indentation.

It is shown that a better combination of strength and fracture toughness results after high temperature heat treatments, but for the uncoated coarse microstructure the biaxial strength depends on the applied loading rate and their resistance to low temperature ageing is severely reduced. By contrast, the coated Y-TZP with the alumina layer has also an excellent combination of strength and fracture toughness, it is more resistance to low environmental subcritical crack growth, and it is inert to degradation after more than 750 hours at 250 ºC in air.

Creep Behavior of a Carbon-Derived Si_3N_4/SiC Nanocomposite

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The creep behaviour of a silicon nitride - silicon carbide nanocomposite prepared by in situ, utilizing $C+SiO_2$ carbothermal reduction is investigated at the temperature from 1200°C to 1450°C in bending under stresses ranking from 50 to 150 MPa in air. The stress exponents are in the interval from 0.8 to 1.28 and the apparent activation energy is 353 kJ/mol. No cavitation was observed by TEM of crept samples and the grain-boundary sliding accommodated by diffusion through the intergranular glass phase is considered as the main creep mechanisms. The significantly higher creep resistance of the nanocomposite compared to the creep resistance of the monolithic silicon nitride prepared with the same amount of sintering additive can be explain with the presence of the intergranular SiC nano-sized particles which limit grain boundary sliding and by the improved viscosity of the intergranular phase due to its changed chemical composition.

Some Expriences of Tribological, Microstructural and Mechanical Investigations of Si_3N_4 Ceramic

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The paper aims at contributing to the better understanding of the tribological behaviour and the damage process during wearing of Si_3N_4 ceramic materials.

Post heat treatment operation in oxidizing atmosphere were applied at different temperatures, (800, 1000, 1200 and 1400°C) on Si_3N_4 based ceramics produced by sinter-HIP method. The wearing behaviour was characterised by the worn volume, friction coefficient, as well as by the geometry of the worn profile after pin-on-disc test. The wear rate showed variation with temperature of the heat treatment.

Microstructural investigations by SEM technique and X-ray diffraction revealed some possible explanation of these changes. These findings were completed by mechanical tests results. Among the possible factors influencing the tribological performance hardness, E-modulus, bending strength and fracture toughness were considered. The correlation of these mechanical properties and the wearing rate is discussed.

The importance of understanding the wearing damage process at microscopic level and identification of the controlling mechanisms are emphasized.

Finite Element Method (FEM) Stress Calculation in a Ceramic Roll

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Due to its outstanding combination of properties like high hardness, fracture toughness and strength, wear resistance and low specific weight, silicon nitride is a well suited material for efficient rolling tools.

In comparison to conventional Hard Metal rolls (Carbides and Nitrides in Metal Matrix) silicon carbide rolls can reach higher service times and are easier to handle because of the lower weight. This is the case for most of the rolled wire materials.

For the processing of high strength materials though, such as Nickel-Base-Alloys, the appearance of cracks was found after short employment which limits the usability of the ceramic rolls.

For a possible solution of this problem, the stresses in the roll had to be investigated by use of numerical methods. The knowledge of the stress level and the stress distribution can lead to an explanation of the observed failures and subsequently help to develop possible countermeasures, so that the ceramic rolls reach the ability to process the whole material range.

Mechanical Properties of Glass Coatings on Ti6Al4V for Biomedical Applications

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One of the solutions to improve osseo-integration of titanium alloy implants, is to coat the piece with a bioactive glass. Because of the thermal expansion mismatch between both materials, a collection of glass layers with different silica content, and hence different thermal expansion coefficients are intercalated in the system in order to improve structural integrity. In this presentation, we present the preliminary mechanical studies done in titanium alloy coated with a glass coating (non-biaoctive), which will serve as the above mentioned intermediate layers. Several glass compositions are studied with indentation techniques, bending, and contact fatigue in air. Results show good structural integrity of the glass and good adhesion to the susbtrate.

Strength Characterisation of Small Ceramic Discs using the Ball-on-Three-Balls Test

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The ball on three balls (B3B) test is a simple, robust and cheap method to evaluate the strength of brittle materials. It has several advantages compared to the common three or four point bending tests, e.g. the test results are not influenced by edges or small geometrical inaccuracies of specimens and of test assembly, the specimen preparation is – in many cases – much cheaper. Furthermore the B3B test assembly has a large capability for miniaturisation.

In a recent paper the relationship between the experimental parameters (applied load, specimen and loading geometry, materials elastic properties) and the stress state has been determined. In this paper the B3B-test is used to measure the strength of an alumina ceramic on discs of different size. A pronounced size effect on strength is observed (the strength of large discs is smaller than that of small discs), which can be explained within the framework of the Weibull theory.

Thermal Shock Behavior of $BaTiO_3$ PTC-Ceramics

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For the modelling of the thermal shock behaviour of components it is assumed in general that the material parameters are independent on temperature. For many materials this is approximately true, but in the case of PTC-ceramics, the relevant material properties are strongly temperature dependent and the above approximation is not possible. The consequences for the thermal shock behaviour are analysed in this paper.

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ESIS Meeting University of Leoben, May, 8th-9th , 2003

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