UNIVERSITY OF WOLLONGONG AUSTRALIA

Thesis writing

Methods

ENGINEERING EXAMPLE

Example: methods section in a thesis (excerpts - part of literature review chapter)

2.8 Summary

final section of lit review, explaining history of experimentation;

In recent years there has been much interest in the development of microwave sintering processes for ceramic materials. Most research has focused on common oxide ceramics, such as alumina and 8 mol% yttria zirconia. Various claims have been made about the potential for the production of oxide ceramics using microwave sintering which possess high density, but smaller grain size than is possible during conventional sintering. However, in some cases, larger grain sizes were found in the microwave sintered materials. Examination of the published literature indicates that very little microstructural work has been performed, and these accounts are all based on the measurement of grain size in only a few specimens at the completion of densification. Direct comparison of the results from various papers is not possible, due to the wide variation in materials and procedures used. No systematic study of microstructural development during microwave sintering has yet been reported.

similar to introductions in published research papers

In the literature there seems to be an underlying general assumption that in microwave sintering the same benefits will be derived for all materials. As there is not yet any model that can explain how microwaves interact with ceramic materials, such an assumption may not be valid.

The aims of this project were to develop a simple, reproducible method for the microwave sintering of two different yttria-zirconia ceramics, and to then undertake a systematic comparison of the microstructural development of these ceramics during microwave and conventional heating using identical thermal profiles. The two materials selected for study, 3 and 8 mol% yttria zirconia, were chosen because although chemically similar, they differ greatly in vacancy concentration, and in densification and grain growth behaviour. In the light of the proposed mechanisms for the conversion of microwave energy to heat, and the reported benefits of microwave sintering, it was considered that these two materials would provide a good indicator of the validity of some of these claims. The effects of heating rate and yttria content were studied during constant rate heating. Isothermal ageing experiments were also performed ...

3.0 Experimental Procedure

a 'preview' would make the text easier to read – ie before presenting details of each technique, first indicate categories covered

3.1 Development of the Microwave Sintering Method

3.1.1 Microwave Equipment

All the initial experimental sintering work was performed in a commercial Sharp microwave oven, model R 2370. This is a 1300 watt, 2.45 GHz multimode chamber with a mode stirrer.

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The true power level could not be altered, but a change in the level of power input over a period of time could be achieved by choosing one of the so called 66 "power levels" from the control panels. These cause the magnetron to operate intermittently, so that a 70% power level, for example, actually means that the samples were exposed to brief periods of full power, interspersed with periods when the magnetron was off. These on-off cycles for the various power level settings are listed in Table 1.

Table 1: Power Cycles for Sharp 1.3 kW Microwave Oven

Power	10%	20%	30%	40%	50%	60%	70%	80%	90%	100%
Time	6	8	12	16	18	22	24	26	30	32
Time	26	24	20	16	14	10	8	6	2	0

details development of simple, reproducible method for microwave sintering

Although it was found to be possible to achieve high density using this oven, the cyclic operation of its magnetron caused a number of problems. Firstly, it meant that the samples were subjected to intermittent heating and cooling cycles over which there was little control, rather than a steady, controlled heating rate. Secondly, it made temperature measurement and control impossible, as the sample temperature fluctuated rapidly through a broad range in response to the changing field. It was also found that there was a plateau in the temperature as a function of time. The maximum temperature attainable was a function of material type, load size, and sample insulation, as well as the power level being used.

Attempts were made to measure the temperature of the samples during sintering using an infra-red pyrometer. A sight hole was made in one wall of the oven, and through the insulation. Problems were encountered with this technique. Firstly, the emissivity of the samples changed as a function of temperature, introducing significant uncertainty as to the reliability of the measurements. Radiation from the susceptors may also have affected the measurements. Secondly, there was considerable heat loss through the sight hole, causing non-uniform sintering of the samples.

3.2 Conventional Sintering

details method used in conventional sintering

Five samples were sintered in each run, in the arrangement shown in Figure 22. The furnace was resistance heated using Crusilite SiC elements located in the roof of a small alumina fibre board lined chamber. A calibrated type R thermocouple connected to a programmable controller was used to control temperature. The thermocouple tip was in contact with the surface of one of the samples. Measured temperature was controlled to within ±5^{0}C of the set point. To increase the uniformity of sintering, and to avoid contamination of the samples, the samples were raised slightly above the alumina fibre insulation by resting them on zirconia supports.

Figure 22: Sample arrangement in the electric furnace [graphic]

3.3 Materials and Sample Preparation

details materials and methods used

3.3.1 Zirconia Powders

High quality spray dried zirconia powders supplied by Tosoh were used. These had nominal dopant contents of 3 mol% and 8 mol% Y203. Details of the chemical composition and particle size, as provided on the manufacturer's data sheets, are given in Table 2. These powders contained small amounts of organic binders to aid in consolidation.

Table 2: Composition and Grain Size of Tosoh Zirconia Powders [*table*]

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3.4 Design of Key Experiments

details methods used in comparative study

Initial work in the commercial microwave oven served to show that it was possible to sinter zirconia to high density while maintaining a small grain size. The "power cycles" which were successfully used to densify the powders are listed in Table 3. The design of the power cycles was an iterative process, with the results of previously used cycles forming the basis for modifications. Early work, which is not included in Table 3, showed that problems with thermal runaway were encountered whenever the 100% power level (magnetron on all the time) was used. The provision of brief periods without the electromagnetic field, such as occurred when using the 70 - 90% power levels, was found to be beneficial in avoiding thermal runaway and sample deformation. Some extended cycles were included to allow grain growth to occur.

However, there was not sufficient control over these experiments to allow any reliable assessment of the effects of various parameters on the process, nor to permit comparison of microwave sintering with conventional sintering methods. The custom-built unit, once commissioned, was able to study these relationships. A statistical design was used to permit efficient investigation of relationships between a number of parameters. Repeat runs were used to provide additional information, and to check reproducibility.

...

3.5 Property Measurement

3.5.1 Physical Properties

Green densities were determined by direct measurement. Densities of sintered samples were determined by the Archimedes method using distilled water with 1 % soap solution as the immersion fluid.

3.5.2 Mechanical Properties

Flexural strength was determined by performing four point bend tests on sintered bars approximately 4 x 5 x 45mm in size. An Instron model 4302 was used to perform the tests, with a loading rate of 0.3 nun/min. Toughness of fully densified samples was measured by Vickers indentation using a 30kg load. Samples were polished to a Igm finish, and gold coated prior to indentation. The gold coating increased the reflectivity of the surface, facilitating identification of crack tips. Toughness was calculated using the following equation: ...

3.6 Characterisation and Comparison of Microstructural Development

details comparison of microstructural development of both samples

3.6.1 Scanning Electron Microscopy and Grain Size Measurement Samples were prepared for electron microscopy using standard ceramographic techniques to polish the surfaces to a l pLm finish. Samples were then thermally etched at 1500^{0}C for 3 minutes in a preheated electric furnace. Either a very thin gold or carbon coating was deposited onto the surface. Cross sections of some samples were cut using a Struer's Accutom-5, so that microstructural uniformity could be assessed. Samples were sectioned in either the longitudinal or transverse direction, and grain size measured in a number of locations along the sections. Fracture surfaces were also studied. Grain sizes were determined using image analysis techniques. This involved ...

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