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Influence of the shaping effect on hardness homogeneity by Vickers indentation analysis

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Abstract

In this study, indentation technique (Vickers indentation) has been unconventionally used to evaluate the homogeneity of barium zirconate ceramic samples which have been shaped through different routes. Statistical tools have been used to estimate the correlation which can be established between heterogeneities within the samples and their shaping ways.

Keywords: Hardness; Defects; Perovskites; Statistical analysis

1. Introduction

Mechanical properties of materials are usually determined with the help of classical bending or tensile tests; most of the time samples with specific forms are required. It is not sometimes possible to produce such a specific sample in order to evaluate its mechanical properties. Indeed, some materials are brittle, rare or expensive such that the machining of samples is often delicate or inconceivable. Indentation tests are then considered to be suitable tools to evaluate mechanical properties of materials difficult to shape. Depending on the indenter geometry and the load used, indentations are about a few nanometres in depth and a few micrometers wide. Indentation tests are thus a local probe but they can be used for small size samples.

As a result, several measurements and a statistical treatment are needed in order to characterize with precision the investigated material.

Based on the same approach, this technique should be interesting for examining possible heterogeneity within a given material.

The indentation techniques give only information about the mechanical properties of the sample surface which must be previously well polished.

Mechanical properties for coating layers[1] or laminated composites[2] are determined by indentation tests. Among mechanical properties which can be deduced from indentation tests, the hardness (H) and the fracture toughness (K_c) are the most popular ones.[3,4]

It is also possible to measure the thermal shock behaviour of brittle refractory materials using an indentation-quench method.[5–8]

To evaluate the thermal shock behaviour, samples containing Vickers indentations are submitted to quenching and the growth of the radial cracks is analysed. It is also possible, under certain conditions, to determine the Young's modulus by Knoop indentation,[4] as well as a relationship between hardness and intrinsic mechanical properties.[9]

In summary, indentation techniques are suitable to determine surface mechanical properties of samples which cannot be manufactured in specific size and shape required when classical tests are used (bending or tensile tests).

In this report, Vickers indentations (and the associated hardness values) have been unconventionally used to evaluate (by mapping) the homogeneity of a given sample which has been shaped by different methods.

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Table 1
Relative densities of green and sintered bodies

	Green density (% TD)	Sintered density (% TD)	Sintered open porosity (%)	Sintered closed porosity (%)
SLIP	50	60	39	0
UNIP	56	69	30	1
ISOP	55	69	30	1

Data recorded from Vickers indentations made on samples shaped by different methods have been compared by using successively elementary statistical analysis, mappings of hardness values for each samples and Weibull distributions. Moreover, non-parametric Kruskal–Wallis test has been conducted to check if the shaping process influences significantly the average hardness value.

2. Experimental

A commercial barium zirconate powder (Alfa-Aesar, 99%), which has a median particle size around 1.6 μm ($d = 0.5$), was milled with a planetary grinder (Retsch PM 400\2) using tungsten carbide jar and 3 mm MgO-stabilized zirconia's balls (Tosoh). After mechanical grinding, a median particle size of 0.6 μm ($d = 0.5$) was obtained.[10]

Three different techniques were used to shape the samples

(i) the slip casting—the so-called *SLIP* sample;A powder/water suspension made of (i) solid contents (30 vol.%; 72 wt.%) and (ii) dispersing agent (1 wt.% (PMAA-Dolapix CE64, Zschimmer & Schwarz)) was slip casted in plaster of Paris cylindrical moulds.[11]
(ii) the uniaxial pressing—the so-called *UNIP* sample;This second sample was uniaxially pressed. Powder was slipped into a cylindrical steel mould and a pressure of 200 bar was applied during 2 min. The mould inner surface was lubricated with the help of a magnesium stearate/acetone solution.
(iii) the isostatic pressing—the so-called *ISOP* sample;The powder was poured into a cylindrical PVC mould and pressed at 2000 bar during 2 min.

According to previous study of barium zirconate sintering processing,[10] it has been possible to select a specific heating program to obtain a relatively porous body.

The samples have been then sintered in air by using the following heating program: isotherm temperature, 1350 °C; heating rate, 1 °C/min; soaking time, 2 h; cooling rate, 10 °C/min.

Notice that it is preferable to perform indentation on a porous ceramic in order to not observe 'piling up' or 'sinking-in' of the material around the Vickers indentation. Indeed, during indentation experiments, the material around the contact area tends to deform upwards or downwards where the load is applied. Such surface deformation modes influence the hardness measurements since the true contact area between the indenter and the specimen increases in the case where pilling-up predominates, while it decreases in the event that sinking-in occurs.[12,13] Piling up can be attributed to the residual stresses produced during impression. Pressure produced during indentation is transferred to neighbour cells, but more intensely to those immediately below the indenter.[12] As a result, the indentation picture focus is not easily made.

The densities of the green pellets were calculated from the sample mass and dimension.

The green body density values are summarised in Table 1 as well as the relative densities, open porosity and closed porosity of sintered samples measured using the Archimede method with *n*-butanol as solvent.

Relative densities were based on a 6.242 g/cm^3 theoretical density which has been calculated from $BaZrO_3$ crystallographic data.

Sintered bodies have been fixed in epoxy resin and then polished with silicon carbide paper and diamond paste until a perfect polished surface is obtained. To improve the contrast between the indentation and the surface and to facilitate the picture focus, a thin layer of silver (10 nm) was coated on the polished surface sample with the help of a sputter-coating machine. Preliminary study revealed that a thin silver layer coating does not influence significantly the hardness of the sample.

The dimensions of green, sintered and polishing samples are summarised in Table 2.

Vickers indentations are realized with an Instron Wilson-Wolpert Tukon 2100B Hardness Tester. This tester uses state of the art closed loop load cell technology to apply the test forces. This ensures that the loading force is constant during the test dwell time.

The Vickers indenter is a diamond square-based pyramid with an angle of 136° between faces. The depth of the indentation is

Table 2
Dimension of green, sintered and polishing bodies

	Green diameter (mm)	Green thickness (mm)	Sintered diameter (mm)	Sintered thickness (mm)	Polished diameter (mm)	Polished thickness (mm)
SLIP	20	10	16	8	16	4
UNIP	33	12	22	8	22	4
ISOP	32	14	23	10	23	5