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Structure investigations of commercial zirconia ceramic powder

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Methodology of research

ABSTRACT

Purpose: The microstructure characterization of commercially available zirconia powder was the purpose of this paper. Different methods of structure analysis were applied owing to the complex, multiphase structure of studied material.
Design/methodology/approach: The X-ray diffraction (XRD) and scanning electron microscopy (SEM) investigations were performed on commercial zirconia ceramic material (Metco 202 (ZrO_2 - 20 wt.%Y_2O_3)). The Rietveld method appeared to be very useful in the verification of the qualitative phase composition and in the determination of phase abundance. Hill and Howard procedure was applied for quantitative phase analysis. The parameters of the individual diffraction line profiles were determined by PRO-FIT Toraya procedure. The powder morphology was analyzed by SEM method practical.
Findings: The presence of Y_2O_3 phase besides of monoclinic, tetragonal and cubic forms of ZrO_2 phase was stated. The cubic zirconia phase appeared to be the main component of the sample (68.2 wt.%) whereas the content of Y_2O_3 is the lowest (4.5 wt. %). The crystallite size of all involved phases lies above nanoscale. The images obtained by SEM reveal the spherical shape of powder particles; their hierarchical type of structure is clearly seen. The greater particles contain smaller ones. The shell of spherical particles is composed of distinct patches.
Practical implications: Performed studies enable the detrmination of the relation between the microstructure of commercial powders and their utilizable properties.
Originality/value: The applied, different methods of structure analysis appeared to be very useful in the microstructure analysis of complex, multiphase material.
Keywords: X-ray phase analysis; Electron microscopy; Rietveld method; Toraya procedure; Ceramics

1. Introduction

Zirconia materials owing to their properties are of widespread application [1]. Pure zirconia in equilibrium state exists in three polymorphic form: monoclinic – below ~1170°C, tetragonal – in the temperature range ~1170–2370°C and cubic – above ~2370°C. The properties of zirconia forms of higher symmetry are very often preferable to monoclinic one. Yttrium oxide is usually used as the stabilization component of higher symmetry zirconia [2]. The increasing interesting in the nanotechnology of zirconia materials is noteworthy [3-6].

The X-ray diffraction and electron microscopy methods are of great importance in the microstructure characterization of complex, multiphase materials. The application of X-ray diffraction methods enables not only qualitative and quantitative phase analysis but also microstructure characterization (crystallite size, lattice distortions, dislocation densities, stacking faults and twins probability) [7].

The aim of the present works is the characterization of the commercially available, multiphase zirconia material using XRD (X-Ray Diffraction) and SEM (Scanning Electron Microscopy) methods.

The Rietveld method [8-9] and Toraya procedure [10] were applied as the tools of XRD patterns analysis. Rietveld method

can be useful in the microstructure characterization and also in the verification of the qualitative phase composition [11-15]. The estimation of phase abundance in multiphase material is possible when detailed information on the structure of concerned phases is available [7]. PRO-FIT Toraya procedure enables the determination of profile parameters of the individual diffraction line. The SEM method was applied for the analysis of powder morphology.

2. Material and research methodology

The structure investigations were performed on commercial zirconia material (Metco 202 (ZrO_2 – 20 wt.%Y_2O_3)).

X-ray diffraction patterns were collected using X-Pert Philips diffractometer equipped with curved graphite monochromator on diffracted beam and with the following slits (in the sequence from Cu tube to counter); Soller (2°), divergence (1/2°), antiscatter (1/2°), Soller (2°) and receiving (0.15 mm). The X-ray data collection was performed for 20–150° 2θ range with 0.04° step.

The profile parameters of individual diffraction lines were determined using Toraya PRO-FIT procedure, which applies Pearson VII function for the description of line profiles.

The Rietveld analysis was performed applying DBWS-9807 program that is an update version of the DBWS programs for Rietveld refinement with PC and mainframe computers. The pseudo-Voigt function was used in the describing of diffraction line profiles at Rietveld refinement. The R_{wp} (weighted-pattern factor) and S (goodness-of-fit) parameters were used as numerical criteria of the quality of the fit of calculated to experimental diffraction data. The phase abundance was determined using the relation proposed by Hill and Howard [7]. Morphologies of the powders were analyzed using SEM (JEOL JSM-6480) method.

3. Results and discussion

Analysis of the X-ray diffraction pattern of studied zirconia material reveals the presence of monoclinic, tetragonal and cubic ZrO_2 phases and Y_2O_3 as well (Fig. 1).

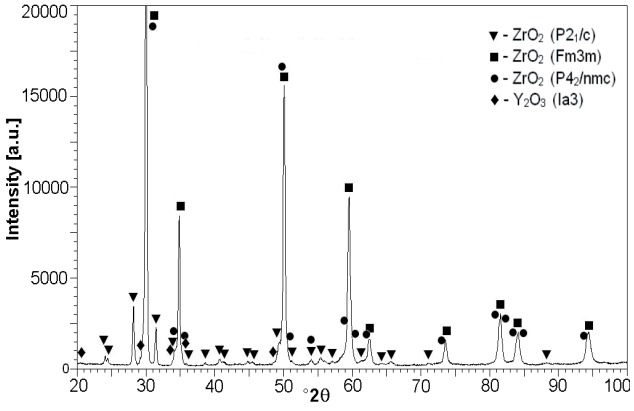

Fig. 1. X-ray diffraction pattern of zirconia sample

Scanning electron microscopy images (Fig. 2) reveal the spherical shape of powder particles. The size of these particles lies in the range from ~5 μm to ~70 μm. From Fig. 2a the hierarchical structure type of greater particles can be postulated; the greater particles contain the smaller ones. Moreover the surface of the particles is rather rough (Figs. 2a,b) and is covered by distinct patches (Fig. 2c).

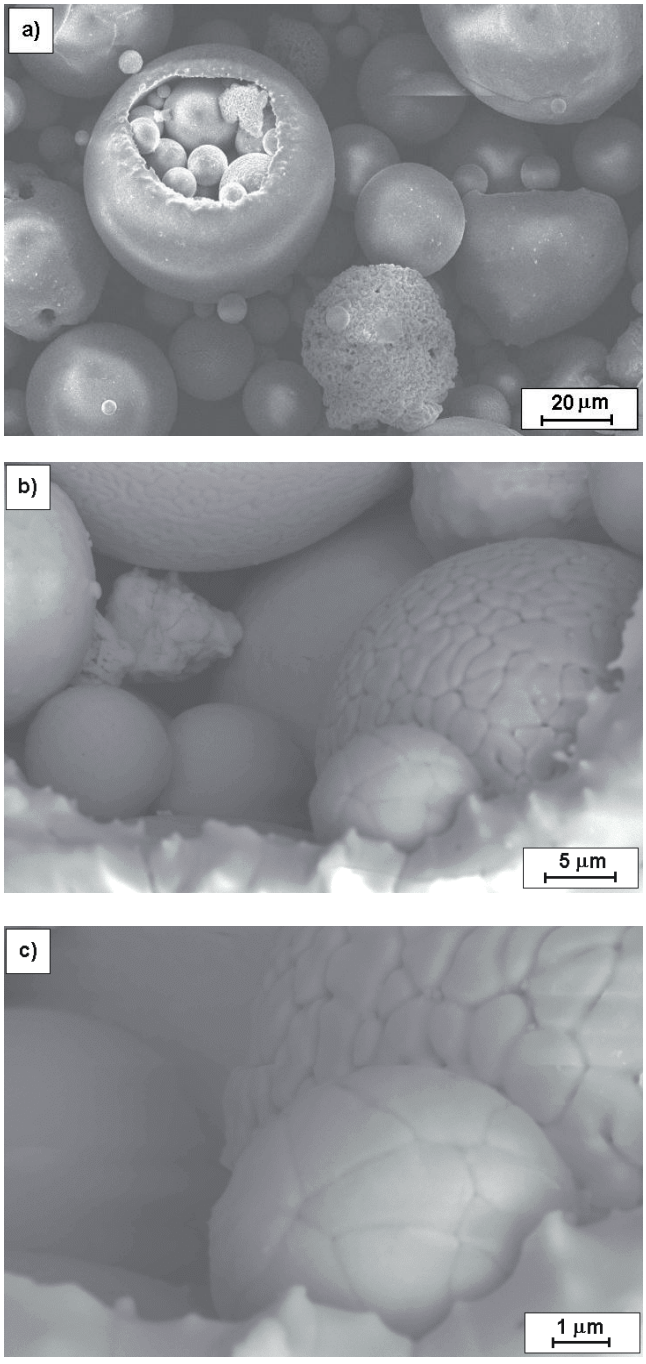

Fig. 2. SEM images of the studied zirconia material

Table 1.
Lattice parameters and the contents of components of zirconia sample

Phase	Space group	Lattice parameters [nm]		Contents [wt.%]
		Rietveld	ICDD	
ZrO_2	$P2_1/c$	$a_0 = 0.51582(8)$	$a_0 = 0.51507(4)$	22.3
		$b_0 = 0.52064(8)$	$b_0 = 0.52028(4)$	
		$c_0 = 0.53184(8)$	$c_0 = 0.53156(4)$	
		$\beta = 99.25°$	$\beta = 99.196°$	
ZrO_2	Fm3m	$a_0 = 0.51490(8)$	$a_0 = 0.5128$	68.2
ZrO_2	$P4_2/nmc$	$a_0 = 0.36667(6)$	$a_0 = 0.35961(1)$	5.0
		$c_0 = 0.52381(8)$	$c_0 = 0.51843(2)$	
Y_2O_3	Ia3	$a_0 = 1.0605(2)$	$a_0 = 1.0604(5)$	4.5

The contents of these phases determined by Hill and Howard procedure [7] are presented in Table 1.

The cubic zirconia phase appeared to be the main component of the sample (68.2 wt.%). The content of monoclinic zirconia phase is still relative high (22.3 wt.%). On the other hand the content of Y_2O_3 phase is the lowest (4.5 wt.%); it means that meaning part of this phase is intercorporated into zirconia phases (nominal content of Y_2O_3 phase is 20 wt.%).

The values of lattice parameters determined by Rietveld method (the accuracy in their determination found using alumina plate SRM 1976 standard is ±0.015%) and these found in ICDD files for all concerned phases are also given in Table 1; generally good agreement between them can be seen.

The Rietveld refinement plot of the sample is presented in Fig. 3. Owing to the presence in the studied material of four phases with one phase of low symmetry, the fitting of calculated pattern to the experimental one seems to be satisfactory.

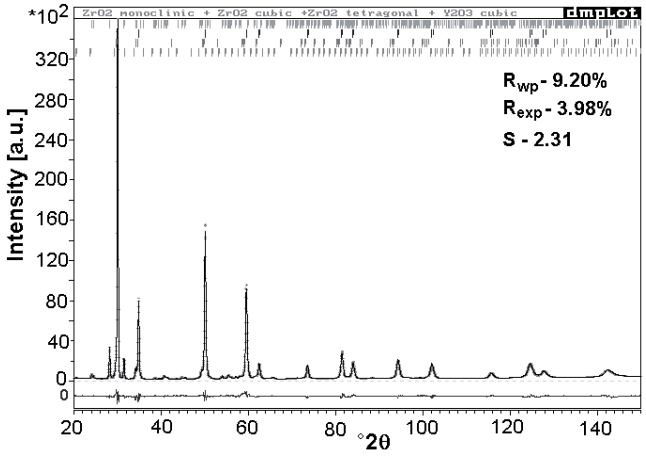

Fig. 3. Rietveld output of X-ray diffraction pattern for zirconia sample

4. Conclusions

- Scanning electron images reveal the spherical shape of powder particles of the size from ~5 μm to ~70 μm. The hierarchical structure type of greater particles can be postulated; the greater particles contain the smaller ones. The surface of the particles is rather rough and is covered by distinct patches.
- The presence of four phases: cubic, tetragonal, and monoclinic forms of zirconia and also Y_2O_3 was stated. It was found that the content of cubic zirconia phase is the highest (68.2 wt.%) whereas of Y_2O_3 one is the lowest (4.5 wt.%). The content of monoclinic zirconia phase is still relative high (22.3 wt.%).
- Good agreement of lattice parameters determined by Rietveld refinement method and these from ICDD files was obtained for all involved phases.
- The crystallite size of all phases found in studied material lies above nanoscale.

Acknowledgements

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References

[1] W. Kurzweg, R.B. Heimann, T. Troczyński, M.L. Waymann, Development of plasma – sprayed bioceramic coatings with bond coats based on titania and zirconia, Bomaterials 19 (1998) 1507-1511,

[2] W.Z. Zhu, Effect of Cubic Phase on the Kinetics of the Isothermal Tetragonal to Monoclinic Transformation in ZrO_2(3mol%Y_2O_3) Ceramics,Ceramics International 24 (1998) 35-43.

[3] U. Betz, A. Sturm, J.F. Loeffler, W. Wagner, A. Wiedenmann, H. Hahn, Low-temperature isothermal sintering and microstructural characterization of nanocrystalline zirconia ceramics using small angle neutron scattering, NanoStructured Materials 12 (1999) 689-692.
[4] X. Bokhimi, A. Morales, O. Novaro, M. Portilla, T. Lopez, F. Tzoompantzi, R. Gomez, Journal of Solid State Chemistry 135 (1998) 28-35.
[5] R. Nitsche, M. Winterer, H. Hahn, Structure of nano-crystalline zirconia and yttria, NanoStructured Materials 6 (1995) 679- 682.
[6] J.A. Wang, M.A. Valenzuela, J. Salmones, A. Vazquez, A. Garcia-Ruiz, X. Bokhimi, Comparative study of nanocrystaline zirconia prepared by precipitation and sol-gel methods, Catalysis Today 68 (2001) 21-30.
[7] Defects and Microstructure Analysis by Difraction, Eds. R.L. Snyder, J. Fiala, H.J. Bunge, IUCr Monogrphs on Crystallography 10, Oxford University Press Inc., New York 1999.
[8] The Rietveld method, Edited by R.W. Young, IUCr Monograph on Crystallography Vol. 5, Oxford Science Pub., 1993.
[9] R.A. Young, D.B. Wiles, Application of the Rietveld methods for structure refinement with powder diffraction data, Advances in X-Ray Analysis 24 (1980) 1-23.
[10] H. Toraya, Array type universal profile function for powder pattern fitting Jurnal of Applied Crystallography 19 (1986) 485–491.
[11] L. Pająk, B. Formanek, G. Dercz, Rietveld analysis of intermetallic phases from Ni-Al system. Proceedings of 11th Scientific International Conference „Achievments in Mechanical and Materials Engineering" AMME'2002, Gliwice-Zakopane, 2002, 405-408.
[12] L. Pająk, B. Formanek, G. Dercz, Dispersion analysis of NiAl-TiC-Al_2O_3 composite powder, Proceedings of 12th Scientific International Conference „Achievments in Mechanical and Materials Engineering" AMME'2003, Gliwice-Zakopane, 2003, 723-726.
[13] G. Dercz, B. Formanek, K. Prusik, L. Pająk, Microstructure of Ni(Cr)-TiC-Cr_3C_2-Cr_7C_3 composite powder, Journal of Materials Processing Technology 162-163 (2005) 15-19.
[14] G. Dercz, B. Formanek, K. Prusik, L. Pająk, Microstructure of Ni(Cr)-TiC-Cr_3C_2-Cr_7C_3 composite powder, Proceedings of 13th Scientific International Conference „Achievments in Mechanical and Materials Engineering" AMME'2005, Gliwice-Zakopane, 2005, 99-102.
[15] L. Pająk, B. Formanek, G. Dercz, Dispersion analysis of NiAl-TiC-Al_2O_3 composite powder ground in high-energy attritorial mill, Journal of Materials Processing Technology (in press).