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Nanoscaled grain boundaries and pores, microstructure and mechanical properties of translucent Yb:[$Lu_xY_{(1-x)}O_3$] ceramics

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ABSTRACT

Translucent ceramics of Yb:[$Lu_xY_{(1-x)}O_3$] system doped by ZrO_2 was sintered from nanopowder synthesized by laser evaporation. The relative density of the ceramics was 99.97%, residual pores had sizes from 8 nm to 20 nm, Young modulus was 200 GPa at the applied load of 2000 mN, the microhardness was 12.8 GPa. The grains of ceramics had sizes 1–10 μm, but the thickness of grain boundaries was about 1 nm. The transcrystalline type of the crack propagation was detected in the specially broken ceramics. The results indicated high strength of grain bonds and good perfection of grain boundaries in the studied ceramics but an increased content of pores (higher than 10^{-3} vol.%) and stoichiometry deviation (Lu:Y:O = 0.21:0.79:3) from the required one (Lu:Y:O = 0.25:0.75:3).

1. Introduction

Optically transparent ceramics is considered as an effective alternative to single crystals for wide applications. However, to achieve high optical properties of the ceramics a lot of technological tasks have to be solved: providing only pure cubic structure of polycrystals, perfect intergrain boundaries with the thickness of about 1 nm, content of pores and impurities not higher than

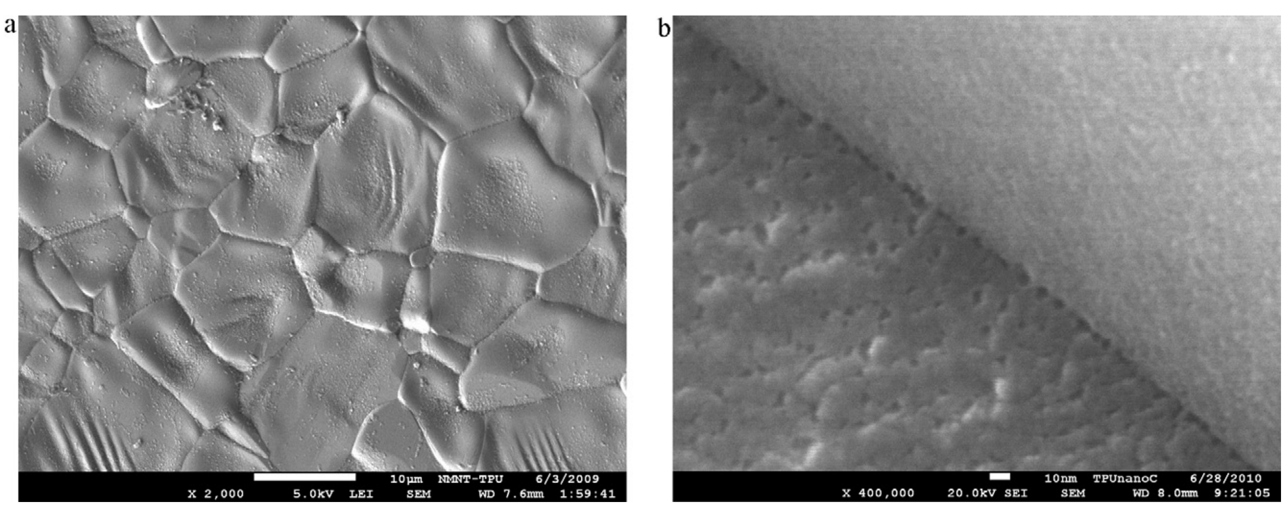

Fig. 1. SEM images of the sintered ceramics surface.

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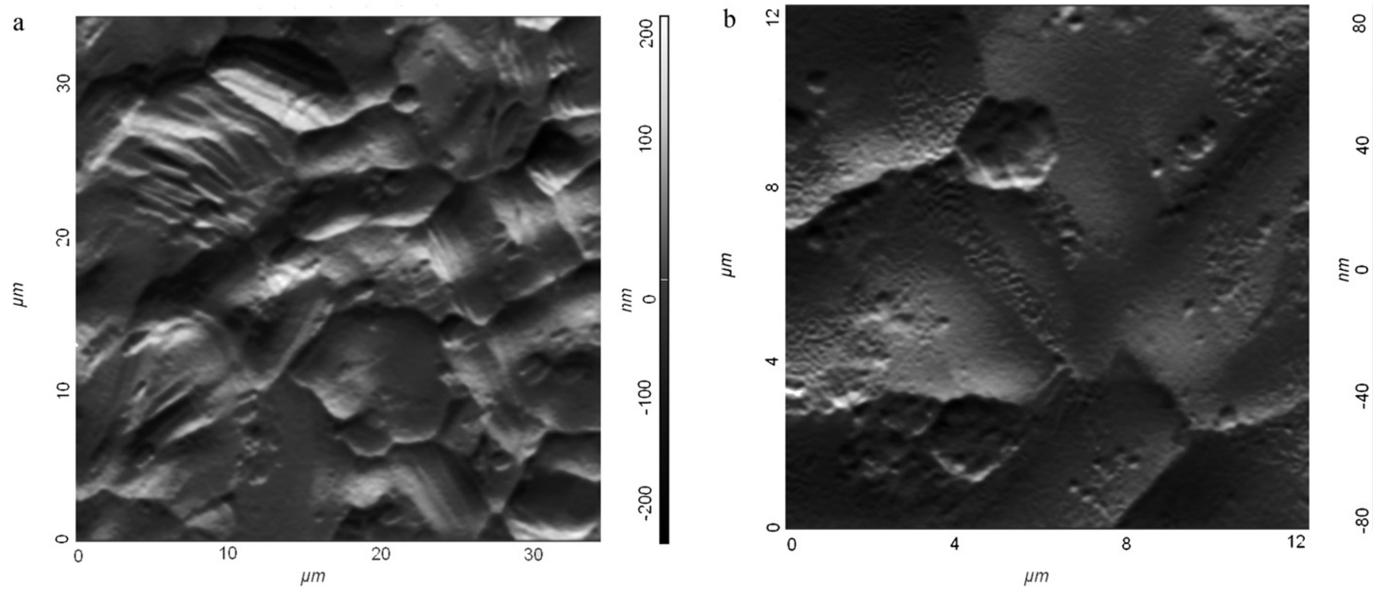

Fig. 2. AFM images of the sintered ceramics surface.

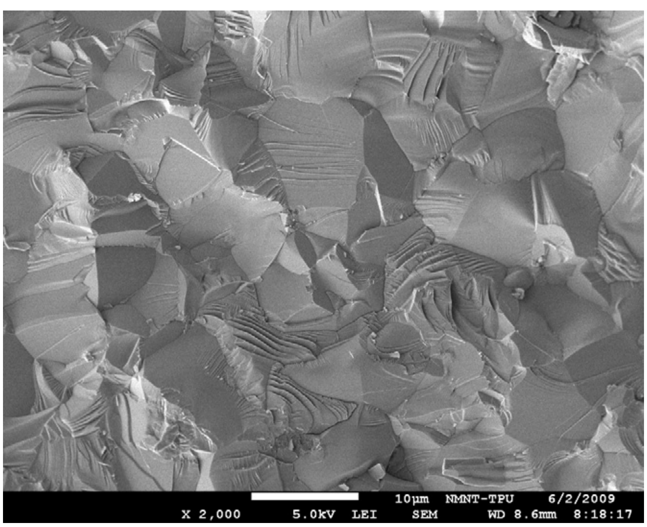

Fig. 3. Spall of the broken ceramics.

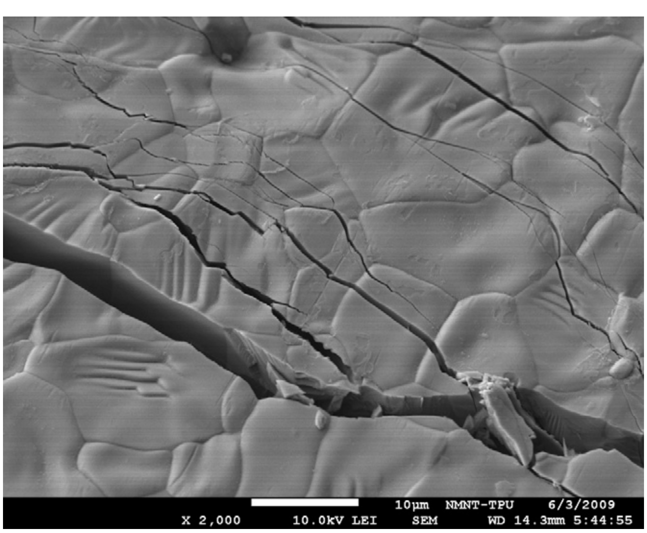

Fig. 4. Transcrystalline propagation of cracks across the spall of the broken ceramics.

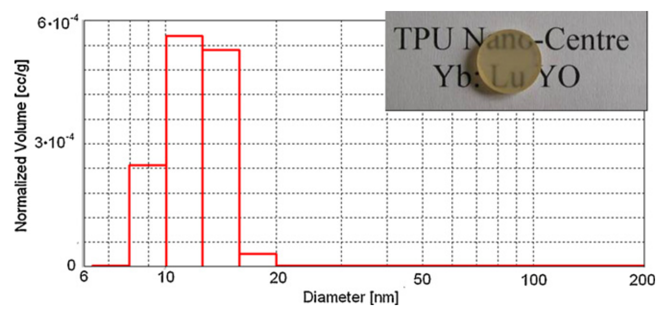

Fig. 5. Pore size distribution in the sintered ceramics. The insert shows the translucent ceramics.

10^{-3} vol.% [1]. So it is important to use a nanopowder (NP) of high quality having the required chemical and phase composition, particle shape and sizes, without agglomerates. But to produce the transparent ceramics the powders should be compacted and then sintered without any defects of microstructure. The stage of powder compaction significantly influences the pore and microdefect content in the sintered ceramics.

In [2] it was shown that a compaction of dry Nd^{3+}:Y_2O_3 nanopowder under powerful ultrasonic action (PUA) allowed to increase the transparency of the sintered ceramics in comparison with the ceramics sintered under the same conditions, but after conventional uniaxial pressing. The aims of this work were both an application of a novel method of powder compaction

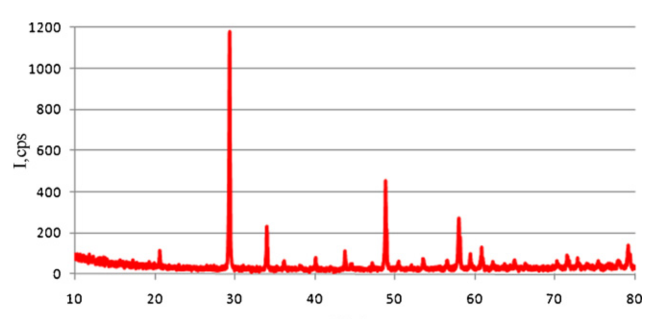

Fig. 6. XRD pattern for the studied ceramics.

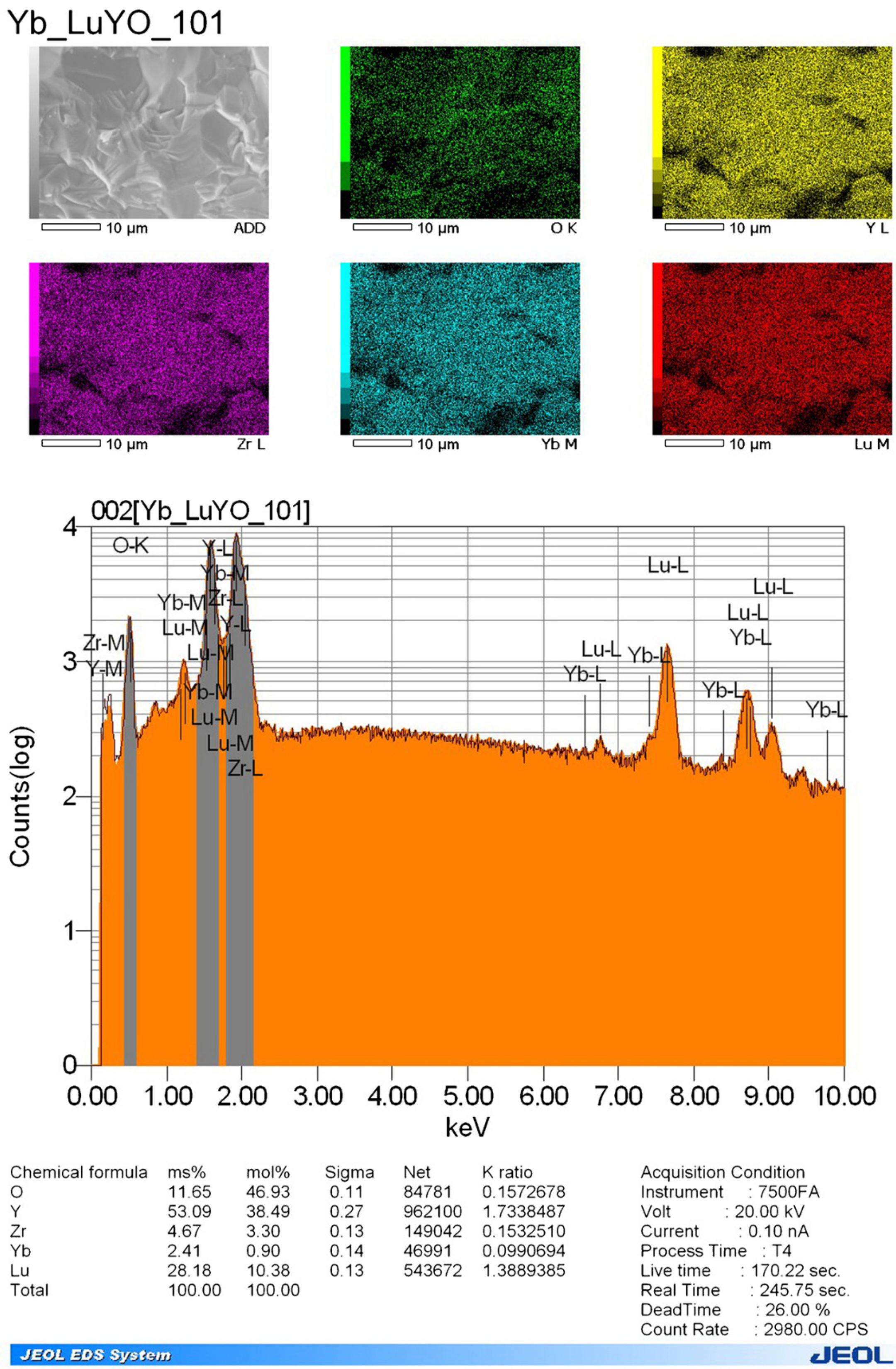

Fig. 7. EDS analysis of the studied ceramics.

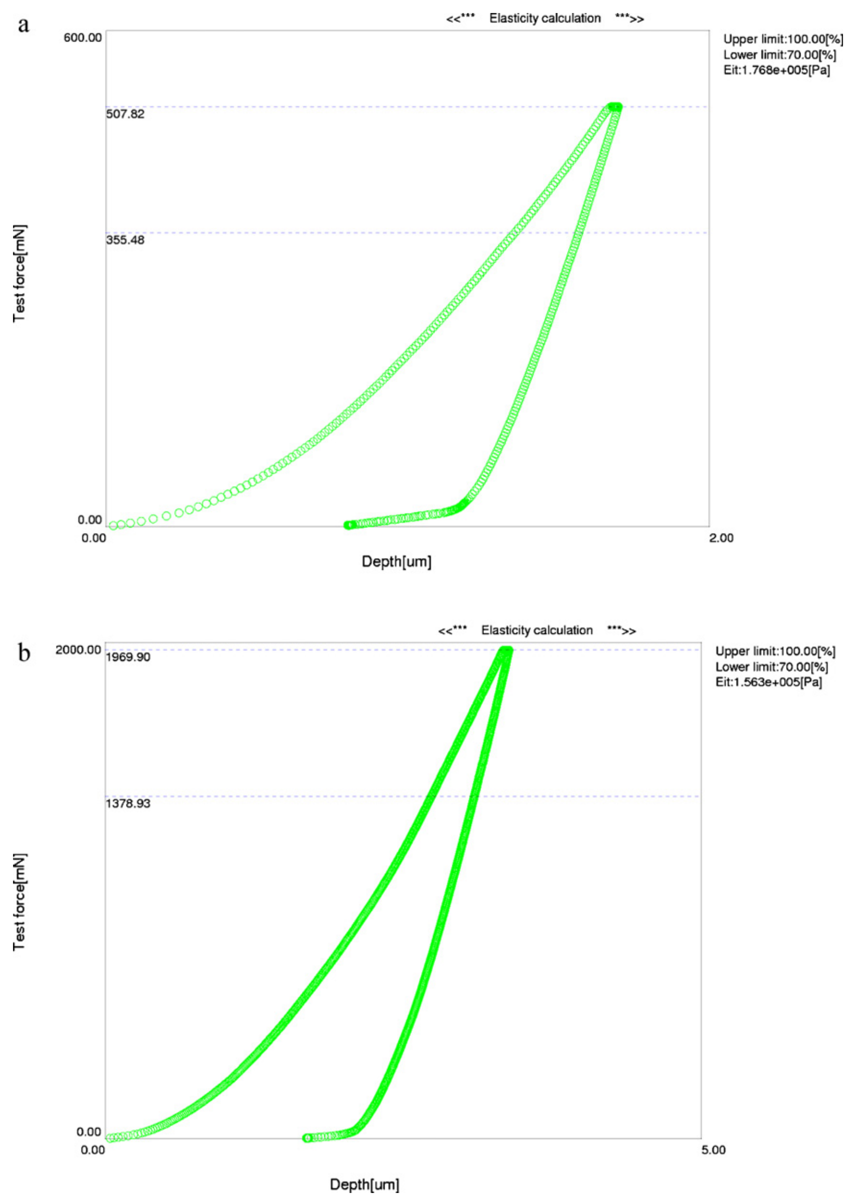

Fig. 8. The plots of the stress–strain curves for nanoindentation of the studied ceramics: (a) load of 500 mN; (b) load of 2000 mN.

under PUA [3,4] for a dry nanopowder with the required content Yb^{3+}:[$Lu_xY_{(1-x)}O_3$] + 6 mol.%ZrO_2 in order to manufacture optical translucent ceramics and a study of microstructure of the sintered ceramics.

2. Synthesis, compacting and sintering of nanopowder

(0.01 vol.%Yb^{3+}):[$Lu_{0.25}Y_{0.75}O_3$] + 6 mol.%ZrO_2 nanopowder with an average size of the nanoparticles of 15 nm was synthesized by evaporation of the target material under focused radiation of a pulsed periodical CO_2 laser and a subsequent vapor condensation in a purified air flow [5].

Pressing of dry NP was carried out by the uniaxial pressing using a hydraulic press WK18 with a powerful ultrasonic action simultaneously supplied to a special die, which was designed as an acoustic wave-guide with a resonant frequency of 21 kHz. Any binders or plasticizers were excluded at nanopowder pressing. Ultrasonic vibrations were supplied from the broad-band ultrasonic generator UZG-6.3 through magnetostrictive transducers PMS-15 to the die transversally to the pressing axis of punches [3,4]. The power of the generator W was 3 kW, the compacting pressure P was 720 MPa. Compacted pellets had a diameter of 14 mm and thickness up to 5 mm.

The pellets were sintered in the vacuum of 1 Pa with a heating rate of 3 K/min at sintering temperature of 1800 °C for 20 h. Then oxidation of the samples was carried out at 1200 °C for 2 h. The sintered pellets were not polished.

3. Results and discussion

3.1. Ceramics microstructure and porosity

The microstructure of the sintered ceramics was studied by the SEM (JSM-7500FA JEOL) and AFM (Ntegra Aura NT-MDT) techniques (Figs. 1 and 2).

The results of the SEM and AFM showed the presence of nearly equiaxial grains ranging in sizes from 1 μm to 10 μm (by the AFM) and with an average size of 8 μm (by the SEM). The thickness of grain boundaries was about 1 nm (Fig. 1b).

To estimate the relative strength of grain bonds one of the ceramics samples was broken in order to investigate the spall surface and also the character of the cracks after a mechanical impact. The analysis of the SEM images presented in Figs. 3 and 4 showed that the breaking of the ceramics had predominantly transcrystalline character: the spall surface consisted of the surfaces of grain fractures (Fig. 3), and the cracks propagated across the grains irrespectively of grain boundary locations (Fig. 4). This fact indicated the high strength of grain bonds.

The pore size distribution in the ceramics was studied using a mercury porosimeter Poremaster 33 Quantachrome (Fig. 5).

It was shown that the residual pores had sizes in the range of 8–20 nm at relative density of ceramics of 99.97% (porosity of 0.03 vol.%), but it was translucent ceramics with low transparency.

According to the XRD analysis (XRD-7000 Shimadzu), the sintered ceramics had the cubic phase of yttrium oxide (Fig. 6).

The results of EDS analysis (JSM-7500FA JEOL) presented in Fig. 7 show the deviation of the ceramics stoichiometry (Lu:Y:O = 0.21:0.79:3) from the required one (Lu:Y:O = 0.25:0.75:3).

So the reasons of low transparency of the studied ceramics were residual porosity higher than 10^{-3} vol.% and non-stoichiometric content.

3.2. Mechanical properties of the ceramics

The elastic modulus and microhardness of the ceramics were measured by the ultramicrohardness gage DUH-211S Shimadzu using a nanoindenter in the form of the Vickers pyramid. The plots of the stress–strain curves are presented in Fig. 8. The Young modulus of the studied ceramics was in the range of 150–200 GPa depending on the applied load of the nanoindenter (500 mN or 2000 mN). The ceramics microhardness was 12.8 GPa. The measurements also showed that the ceramics was characterized by an expressed contribution of a plastic component of deformation, that is, the relative value of a plastic component of deformation work made up about 60% of the total one, and the coefficient of material fluidity under indentation was about 2%.

4. Conclusions

The technique of dry nanopowder uniaxial compaction under a powerful ultrasonic action allowed to sinter translucent Yb^{3+}:[$Lu_xY_{(1-x)}O_3$] + 6 mol.%ZrO_2 ceramics with porosity of 0.03% and pore sizes from 8 nm up to 20 nm. The ceramics had microhardness of 12.8 GPa and high strength of grain bonds with the thickness of grain boundaries of about 1 nm.

To improve the transparency of this ceramics it is necessary to optimize the conditions of compacting and sintering for the purpose of porosity reduction lower than 10^{-3} vol.% and to provide the required stoichiometric content.

Acknowledgements

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