NANOINDENTATION STUDY OF ZnO, ZnO: In, Zn: Ga SCINTILLATION CERAMICS

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Motivation

ZnO is a multifunctional material with a wide range of applications. Recently ZnO ceramics based on nanopowders are of special interest for the use as scintillators with high-efficiency [1].The *introduction of donor impurities such as gallium or indium into the composition increase scintillation characteristics of ZnO ceramics* [2]. For perspective technical use of these doped ceramics no less important role is played by the mechanical properties, including the fracture mode, hardness (H, GPa) and modulus of elasticity (E, GPa).

In this research the ZnO undoped and doped with optimal for luminescent properties concentration ZnO: 0.13 wt% In and ZnO: 0.075 wt% Ga ceramics, obtained by hot pressing in vacuum [2] have been investigated.

Experimental

The fracture mode was studied by scanning electron microscopy (SEM). Mechanical properties of ceramics were characterized using nanoindentation method. The data obtained allow not only hardness but also Young's modulus and work of plastic deformation to be derived. The Berkovich diamond indenter (tip radius <20 nm) was used for direct continuous loading and displacement registration. To understand the distribution of In and Ga, topographic measurements of nanohardness were carried out both near the grain boundaries (GBs) and at centers of grain.

Results and discussion

In Fig.1 the data of hardness (a) and elastic modulus (b) *vs* displacement into surface (h,nm) for ZnO undoped ceramics are presented. At h= 100 nm,the hardness value is H= 3.0 GPa and decreases gradually to 2.6 GPa at h= 1μm, that could be connected with the influence of GBs.

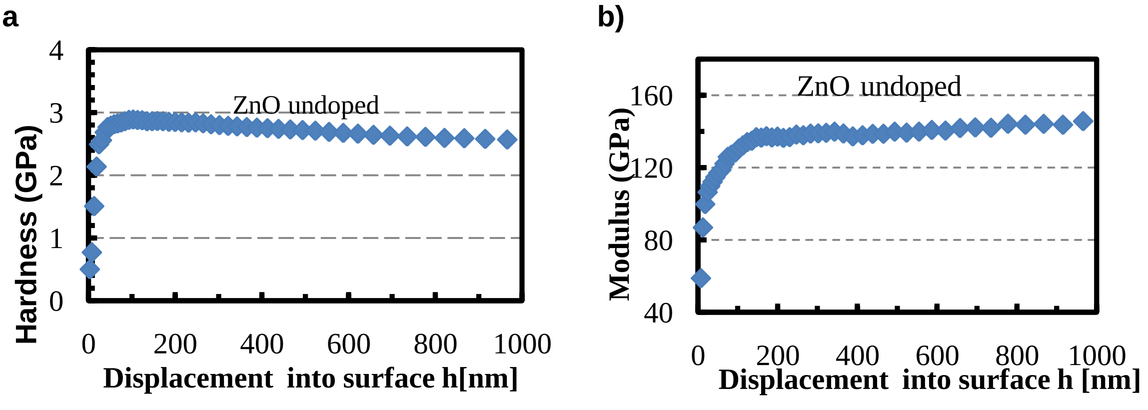

Figure 1 - Hardness (H, GPa) andYoung modulus (E, GPa) of ZnO undoped ceramics.

The effect of boundaries on hardness in ceramics is often associated with the brittle fracture along boundaries. We estimated the ratio of the size of deformation zone around the imprint with the grain size at different indentation depths (h) [3]. As is known, according to the Berkovich hardness, formula $H= 2.092\,\frac{P}{a^2}$, where *P* –load, *a*- side of indenter imprint, *h* is calculated as *h*= *a*/7.7. The size of the deformation zone (*t*) around the imprint we can calculate as $t = n \cdot a$, accepting the coefficient *n* equal to *n*=1.5 for oxide systems. As an example, let as consider the hardness of undoped ceramics shown in Fig. 1, that has grain size in range d_g = 8 - 25 μm. At indentation depth *h*= 100 nm, *a*= 770 nm, obtained *t*= 1.1

µm, hence at *h*≤100 nm deformation zone is within the grain and the measured hardness (3.0 GPa) corresponds to hardness of a grain. Note that this value is normal for ZnO crystal. In case when the size of deformation zone exceeds the size of grain, the hardness can be affected by properties of GBs.

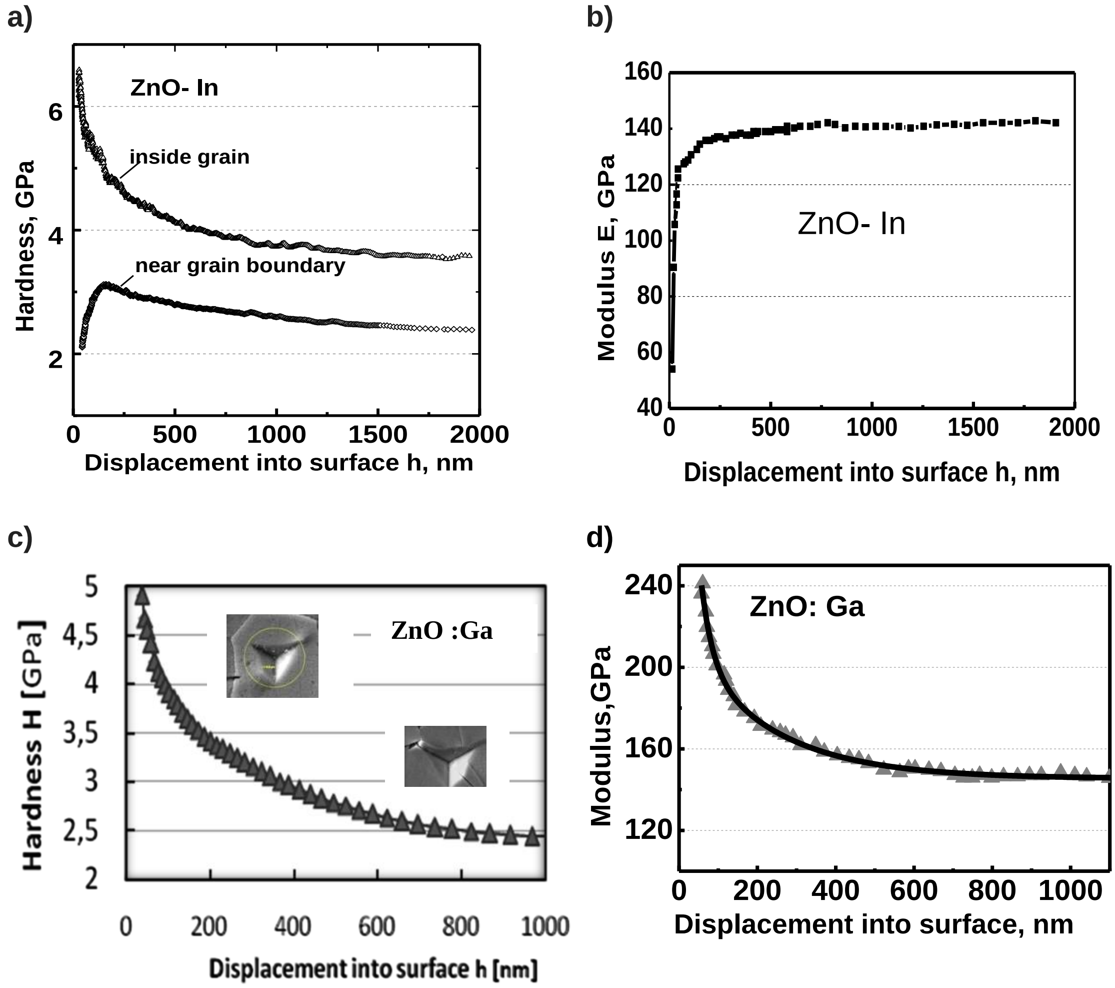

Figure 2 - Hardness H, GPa and Young modulus E, GPa of ZnO-In (a,b) and ZnO-Ga (c,d) ceramics.

The results of nanoindentation measurements on ZnO doped ceramics are shown in Fig. 2**.** The nanoindentation on ZnO: In ceramics (Fig. 2 a**)** was carried out both at the centre of the coarse grains and near the GBs, using the technique of topographic indentation "in situ" in the nanoindenter tester. As follows from Fig. 2 a, an interesting result was obtained: indium additive leads to increase of the hardness inside grains (H= 4.5 GPa at 100 nm and 3.8 GPa at the maximum depth), moreover, enhances indentation plasticity near GBs (H= 2.5 GPa, without brittleness). Analysis of loading curves revealed significant increase of the work of plastic deformation from 81 % for grain to 90.5% near GBs. Obviously indium increases the adhesion on GBs and provides continuity of GBs. As seen from Fig. 2b, in ZnO-Ga ceramics (d_g = 8- 25 µm) the hardness inside grains H= 5 GPa and E= 170 GPa, but in the deformation zone including many grains and sub-grains, the hardness was reduced twice, which is associated with the cracking. Thus, gallium does not strengthen the boundaries, but rather contributes to the brittleness. On the other hand, high values of both hardness and Young's modulus with small indentation depths could indicate the effect of gallium on the structure inside the grain.

Analysis of SEM images of fracture surfaces as an important characteristic for sintered ceramics, confirms mainly the brittle intergranular mode of fracture in the case of undoped ZnO ceramics (Fig. 3a). Fracture of ZnO ceramics doped with In has

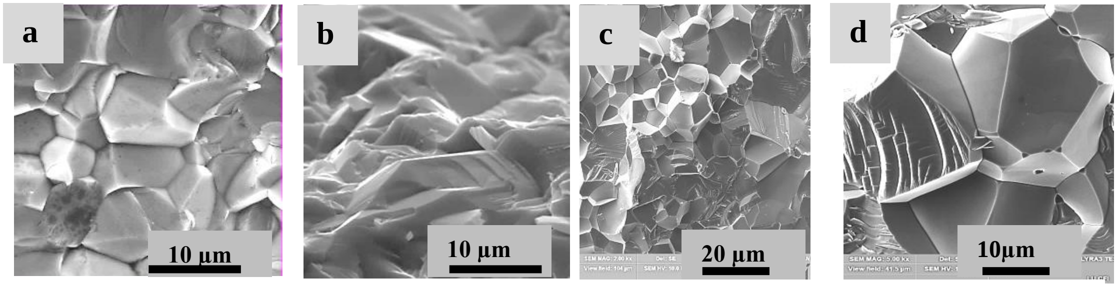

Figure 3 -SEM images of ZnO ceramics fracture: undoped (a), In doped (b) and Ga doped (c,d) ceramics.

transcrystalline character without the presence of substructure and micropores (Fig. 3b). The microstructure of fracture surfaces of ZnO:Ga ceramics has a completely different character compared with In doped ceramics. Fracture demonstrates a brittle intercrystalline mode (Fig. 3c,d). The presence of the substructure and micropores on the GBs is revealed (Fig. 3d).The results of fractographic research are in agreement with the data of nanoindentation for investigated ceramics.

Conclusions

Comparative analysis of the results shows that doping with both indium and gallium has a significant effect on the mechanical properties of ZnO ceramics. However, the mechanisms of action are different. ZnO:Ga ceramics has intergranular brittle fracture mode and low crack resistance (toughness) by nanoindentation. On the contrary, ZnO: In ceramics is characterized by transcrystalline fracture mode, increased of hardness and plasticity near GBs. Results obtained give evidence, that ZnO:In has a greater mechanical stress relaxation potential than ZnO:Ga ceramic, that is very important for the use of ceramics as scintillator.

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References.

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