MECHANICAL CHARACTERISATION OF ZIRCONIUM OXIDE THIN FILMS DEPOSITED BY CHEMICAL VAPOR DEPOSITION

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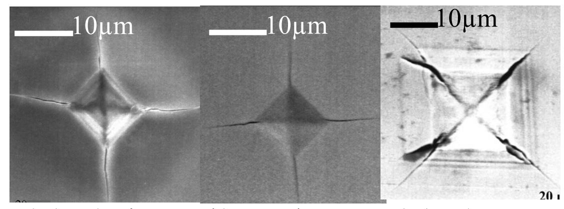
Zirconia (ZrO_2) thin films were deposited by Metal Organic Chemical Vapor Deposition (MOCVD) on various substrates such as (100) Si and (001) $SrTiO_3$ single crystals, with or without $YBa_2Cu_3O_7$ as an interlayer. The deposition process and characteristics were described in a first paper. The film cristallographic structure and microstructure as observed by TEM (transmission electron microscopy), FEG and AFM were also presented in the same paper. The aim of this contribution consists in characterising the mechanical behavior of the zirconia films, according to the nature of the substrate. This was performed using Vickers micro-indentation and Berkovitch nano-indentation tests on 150 nm (on Si) or 80 nm (on STO) thick ZrO_2 films.

Even with small loads, the Vickers micro-indentation tests did not allow to determine quantitative features due to the important penetration depth compared the film thickness. But, as in most cases, damages are created around the prints (Figs.1), qualitative information about the damage criterium can be obtained: in all cases, microcracks are observed aligned along the dense (111) planes of the substrate, a strained area is often observed around the print (Figs.1a, c, d) and spalling occurs (Figs.1d,f). From the difference in length between the cracks formed on the bare substrate and those on the covered substrate, a toughness classification can be suggested. Moreover, information on the film adhesion can be deduced from buckling and spalling density.

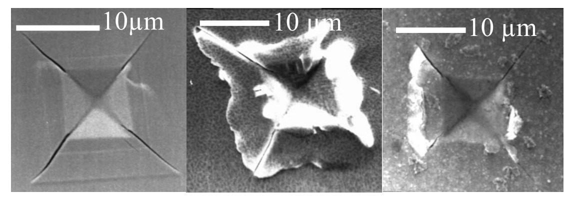
In order to obtain quantitative data on the substrate and film hardness and their Young modulus, nano-indentation tests were performed. Images of the prints observed by SEM are given in Figs.2. In these cases no damages are visible. Values of hardness and Young modulus determined using nanotests are gathered in Table I. In Fig.3, the evolution of the hardness according to the inverse of the penetration depth of the indenter shows that hardness tends towards the film hardness only when the penetration depth is small (on the right in Fig.3), and towards the substrate hardness when the penetration depth is important (on the left in Fig.3).

It is observed that zirconia films on silicon and on STO have relatively good cohesion and adhesion (see Figs.2a and b, while zirconia films on YBaCuO/STO are not really satisfying (Fig.2c). This is opposite to previous results obtained on (Pb,La)TiO_3/YBaCuO /STO system (1) where the YBaCuO interlayer had a good influence on the mechanical properties of the system.

1. O. Bernard, S. Poulat, A.M. Huntz, M. Andrieux,, S. Poissonnet. Inter. Conf. on Thin Films Deposition of Oxide Multilayers. Hybride Systems, TFDOM 2, 18-19 oct 2001, Autrans, F. EDP Sciences, ed. J. Kreisel et C. Dubourdieu, J. Phys.IV, vol.11, 35-39.

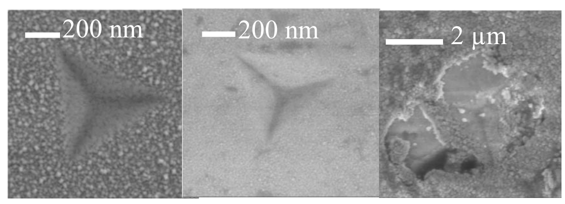


a) (100) Si b) ZrO_2/STO c) (001) STO



d) ZrO_2 150 nm on Si e) ZrO_2 150 nm on STO f) ZrO_2 on YBCO/STO

Figs.1: Vickers prints (a-d) 1N and (e-f) 0.5N load



a) ZrO_2 (150 nm) on Si b) ZrO_2 (80 nm) on STO c) ZrO_2 on YBCO/STO

Figs.2: Berkovitch nanoprints prints (a-b) 200nm penetration depth and (c) 1000nm penetration depth.

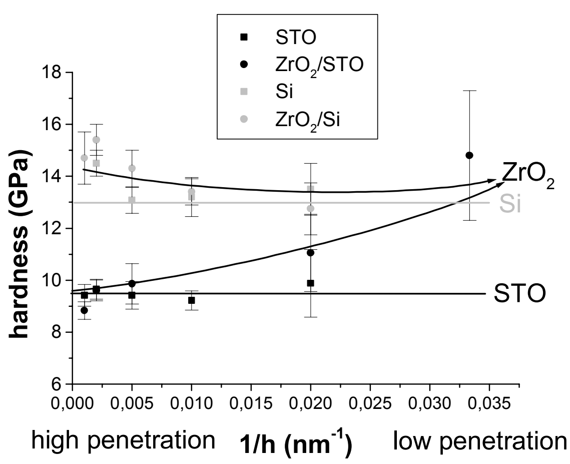


Fig.3: Evolution of the hardness vs 1/penetration depth.

Table I: Values of mechanical characteristics determined by nano-indentation tests.

Materials	H (GPa)	E (GPa)	E/H
Si	13	180	13.8
ZrO_2/Si	13	225	17.3
STO	9.5	225	23.7
ZrO_2/STO	13	220	16.9
YBaCuO/STO	12.5	235	18.8
ZrO_2/ YBaCuO/STO	un-detected	un-detected	un-detected