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Nano-microscale Characterizations to Al_2O_3/SiC_{nano} Ceramic Composites

Zhong Ling

State Key Laboratory of Nonlinear Mechanics, Institute of Mechanics, Chinese Academy of Sciences, Beijing 100080, China, Email: lingz@lnm.imech.ac.cn

Abstract

In this paper, the principle physical properties, Young's modulus and hardness at very small volume, are measured via nanoindentation for one typical nano-ceramics composite. The Young's modulus and hardness presented in this paper are dependent obviously on components or microstructures of the current material while penetration is at 10~100nm. With the penetration getting deeper, the final magnitude of the modulus seems to be a certain one for the composite. Compared with the modulus, the hardness seems to be more sensitive to the penetration depth. Microscopic observations are also performed under optical microscopics to make sure that, at very tiny volume, the nanoindentations could be local at components of the composite and the final results obtained from nanoindentation tests.

Key words: nano-ceramic composite, nanoindentation, components, physical property

1. Introduction

Ceramics possesses high refractoriness, good wear resistance and chemical stability. For such "toughened" materials, however, the central technical problem is their lack of ductility. That is, these materials have no mechanism for redistributing stress; therefore, strain concentration sites are also regions of high stress and small fracture resistance [1]. One attempt to alleviate stress concentration and gain improved toughness and ductility is to design ceramic matrix composites (CMC_s) by dispersing second particles in their matrix.

Nano-composite ceramics is one of ceramic composites based on such reinforcing mechanism. Previous investigation showed that, nanosized particles dispersed within grains of matrix would lead to a change in the fracture mode, from predominantly inter-granular to predominantly trans-granular fracture and, nanosized particles existed on grain boundaries would link with boundary strength [2-4]. In these nanocomposites, the ceramics grains are microns (<10 microns) and the second phase particles are nanometers (<100nanometers). Obviously, the stress-redistribution in the grains and particles' reinforcing mechanism to the grain boundaries in the nanocomposites are either microscale or nanoscale and strongly dependent on the components' dispersion and their scale compatibility. For such nanoceramic composites, it is more essential and important to better understand the physical and mechanical properties of each componets of the composite. However in the past years, few research reports are appeared on nano- micro- scale quantitative description for the essential mechanical behavior of nanocomposites due to limited methods for micro- or nano- scale measurement and observation.

Nanoindentation is a technique developed over the last decade for probing the mechanical properties of materials at the smallest possible scale. Many researches are done on the nanotechnique's mechanisms and applications to measure mechanical properties at tiny volume of material [5]. In this paper, taking the advantage of the nanoindentation techniques, the principle physical properties, Youngs' modulus and hardness, at very tiny volume are measured for a typical nanocomposite, Al_2O_3/SiC_{nano}. The microscopic observations are performed carefully to make sure each indentation location. Finally the Young's modulus and hardness are described quantitatively for each component in the current composite.

2 Material & Test Procedure

Material in current work is Al_2O_3/SiC_{nano}, in fact, the matrix is consisted of Al_2O_3 and some ZrO_2, Al_2O_3:ZrO_2=92:8. Volume fractor of SiC particles in whole composite is about 10 vol.%. The Table 1 shows the principle parameters of the composite. Figure 1 is the micrograph of original material and three components, Al_2O_3 and ZrO_2 and SiC, could be seen.

Table1 Main parameters in the composite

component	Al_2O_3: ZrO_2	SiC
fractor	92:8	10vol.%
Size (μm)	5	0.1

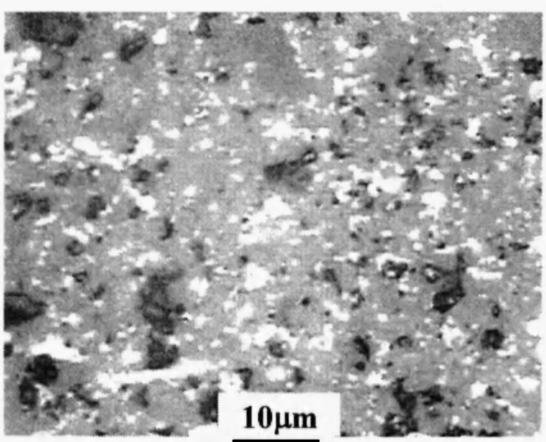

Figure1.Micrograph of Original material.

Nanoindentation tests are performed under nanoindenter, ***Nano Indenter XP***, with Berkovich tip, pyramid diamond; the maximum penetration depth is set as 300nm, the loading rate 0.1nm/s.

Optical microscopy, ***Polyvar-met***, with image-system, is applied to make sure the micrograph of each nanoindentation and its location.

3. Experimental Observations

3.1 Nanoindentation test data

Total three groups of nano-tests were performed. Table 2 shows two groups of the test data. Their penetration depths are about 310nm and the maximum loads on the nanoindenter for each indentation vary a bit, so do their young's modulus and hardness. (see Table2)

Table 2 Test data & results(Dep.=300nm)

Pt_#	P_m(mN)	D_{max}(nm)	E^*(GPa)	H^*(GPa)
n3_1	37.51	309.70	399.62	24.31
n3_2	38.79	310.45	368.16	26.43
n3_3°	31.00	313.36	348.71	18.10
n3_4°	28.85	310.63	339.81	16.93
n3_5	40.10	309.61	425.81	26.33
n4_1	38.04	312.83	392.13	24.04
n4_2°	27.07	309.96	326.35	15.60
n4_3	38.35	311.30	412.93	24.23
n4_4°	32.35	315.43	350.53	19.01
n4_5	36.51	316.38	376.49	22.10

*The modulus and hardness are read from unloading curves

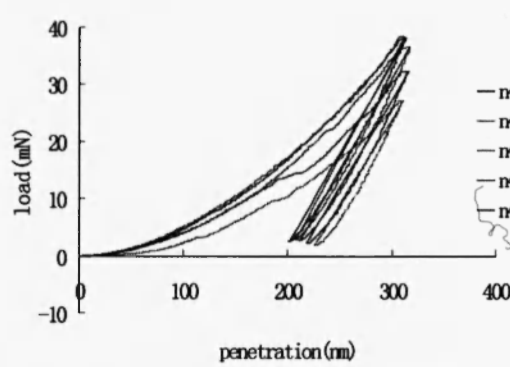

Figure2-a Load process with penetration varied.

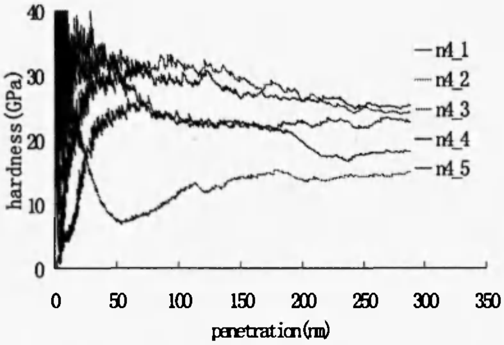

Figure2-b Hardness process with penetration nanoindentations

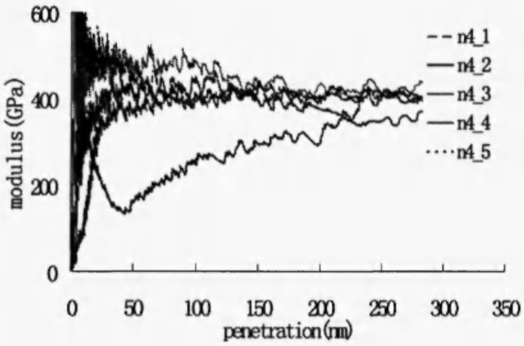

Figure2-c Modulus process with penetration varied

Figure 2 presented typical test curves of load, hardness and Young's modulus gotten in one group of tests (n4, depth=10~300nm). Compared these with the measurement data listed in Table2, the varieties of magnitude of load, hardness and Young's modulus correspond to their traces with penetration depths. The further microscopic observation is needed to check these nanoindentations' location and if these traces contain some information related to physical properties of each component in such heterogeneous material.

3.2 Microscopic observation

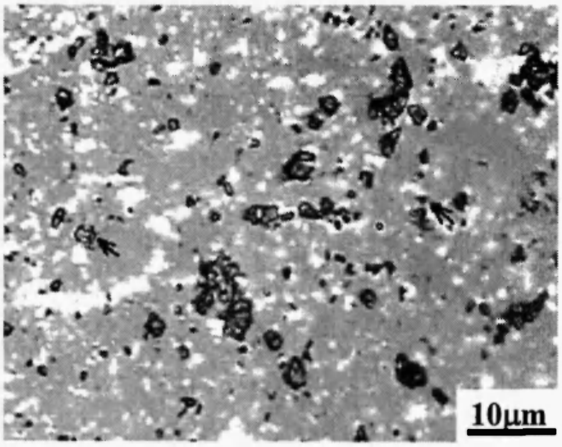

Figure 3-a Micrograph for indentation n4-1&2, where indentation n4-1 is local at white region and n4_2 local at the dark region.

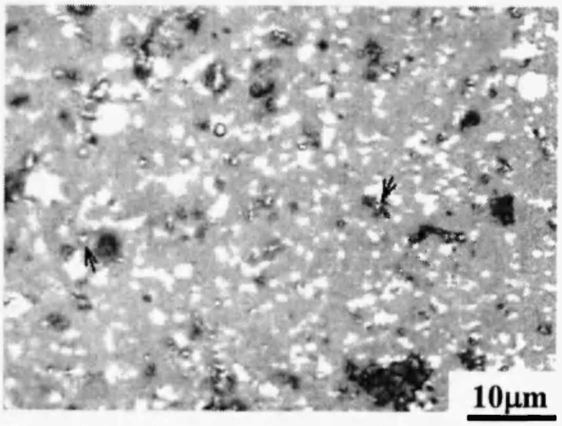

Figure3-b Micrograph for n4_4&5 marked by arrows, n4_4(right) is local at dark region and, n4_5 is at grey region.

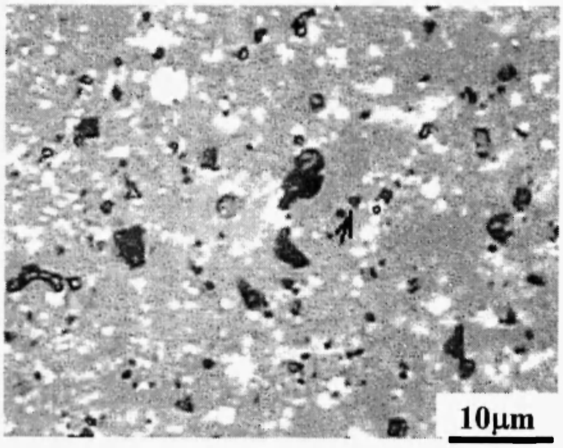

Figure3-c Micrograph for n4_3, which is at white region marked by arrow.

Microscopic observation to these indentations would give the effective traces to understand the phenomena appeared in test data and figure2. In order to get the relation linked test data and real composite, micrographs of group-n4 are chosen and each indentation in the graph is marked distinctly by arrow (see Figure3).

4. Summary

Actually, in whole test results, the main interesting features could be concluded as two typical nanoindentations:

(1) Their hardness and modulus go up quickly while depth at 10-50nm or <100nm, then go down slowly and get to a constant at 100-300nm; their hardness at unloading is about 22-26 GPa, and their Young's modulus about 360-420GPa(see Table2, the samples without superscript "o"). They are almost local at white and grey regions in the micrographs, Al_2O_3 (see Figure3-a, n4_1). It looks to others so, such as samples n3_1, _2, _5, and n4_1, _3 , _5;

(2) For another type of nanoindentations, the hardness and Young's modulus go down quickly while depth 10nm-50nm (see Figure2-b and 2_c, sample n4_2), then go up at depth 50-200nm, after 200nm, they get to a constant. The hardness at unloading is about 15-19 GPa, and the modulus at unloading is about 320-350GPa. Microscopic results to these samples show that, they are all local at the dark region, i.e. ZrO_2 (see Figure3-a, n4_2 and Figure2).

Finally, Table 3 gave hardness and Young's modulus for homogeneous materials measured via nanoindentation [5,6]. Compared with those in Al_2O_3, SiC and ZrO_2, the current results of hardness and Young's modulus, got from each component in heterogeneous composite, are reasonable.

Table 3 some measure results in [5,6]

Material	*E(GPa)**	*H(GPa)**
ZrO_2	310.6±11.2	20.2±1.2
Al_2O_3	474.4±41.3	34.8±4.0
ZrO_2	275.0±10.2	14.3±0.7
Al_2O_3	416.6±32.4	22.6±2.1
SiC	427	21.8

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