

INDENTATION LOAD–SIZE EFFECT IN Al_2O_3 —SiC NANOCOMPOSITES

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The indentation load-size effect (ISE) in Vickers hardness of Al_2O_3 and $\text{Al}_2\text{O}_3 + \text{SiC}$ nanocomposites has been investigated and analysed using Meyer law, proportional specimen resistance (PSR) model and modified proportional specimen resistance (MPSR) model. The strongest ISE was found for alumina. Both the PSR and MPSR models described the ISE well, but the MPSR model resulted in slightly lower true hardness values for all materials investigated. No evidence of the effect of machining stresses on the ISE has been found.

Key words: Al_2O_3 , SiC, hardness, indentation load-size effect

1 INTRODUCTION

During the last years it was frequently reported that the measured hardness increased with decreasing load [1, 2]. To explain this so called “indentation load/size effect — ISE” intensive research has been performed during the last decade, based on which different explanations have been advanced [3, 4]. Several empirical or semi-empirical equations, including Meyer law [5], the Hays-Kendall approach [6], the energy-balance approach [7, 8], the proportional specimen resistance (PSR) model [2], etc. have been proposed for describing the variation of the indentation hardness with the applied indentation load. Probably the most widely used empirical equation for describing the ISE is Meyer law, which gives an expression relating the load (P) and the size of indentation (d) of the form

$$P = Ad^n, \quad (1)$$

where the exponent n , *ie* Meyer index, and A are constant. If $n < 2$ there is an ISE on hardness and when $n = 2$, the hardness is independent of the applied load.

Li and Bradt in their PSR model [2], prepared on the basis of the work in [6], suggested that the specimen resistance W , during indentation is not a constant, as was proposed by Hays and Kendall, but increases with the indentation size and is directly proportional to it according to the relationship

$$W = a_1d \quad (2)$$

and the effective indentation load and the indentation dimension are therefore related as follows

$$P_{\text{eff}} = P - W = P - a_1d = a_2d^2. \quad (3)$$

Gong *et al* [9] suggested a modified PSR model based on the consideration of the effect of the machining-induced residual stresses at the surface during the indentation in the form:

$$P = P_0 + a_1d + a_2d^2, \quad (4)$$

where P_0 is a constant and a_1 and a_2 are the same parameters as in the PSR model.

The investigations up to now concerning the ISE in ceramics have focused mainly on single crystals, monolithic and composite ceramics and only a limited investigation has been carried out on ceramic nanocomposites.

The aim of the present investigation is to study the load dependence of the measured Vickers hardness of alumina — silicon carbide micro/nano composites and to describe the indentation — size effect using different models.

2 EXPERIMENTAL

The experimental materials have been prepared in the collaboration with Department of Materials, University of Oxford, Oxford, United Kingdom. Monolithic Al_2O_3 and Al_2O_3 -SiC nanocomposites with 5 vol% (A5) and 10vol% (A10) of SiC particles were prepared by hot pressing in a graphite die for 30 minutes at 25 MPa in an argon atmosphere at 1700 °C for the nanocomposites and 1550 °C for the pure alumina.

The microstructure and fracture surfaces were observed by scanning electron microscopy. The hardness was determined using Vickers indentation method with applied loads ranging from 1 N to 49.05 N for. The load dependence of the measured Vickers hardness of monolithic alumina and alumina — silicon carbide micro/nano composites has been investigated on different models: Meyer law (1), Proportional specimen resistance (PSR), see (3) and modified PSR, according to (4).

3 RESULTS AND DISCUSSION

Microstructure of the sample Al_2O_3 consists of Al_2O_3 grains separated by grain boundary phase. Increased content of SiC resulted in markedly altered microstructure.

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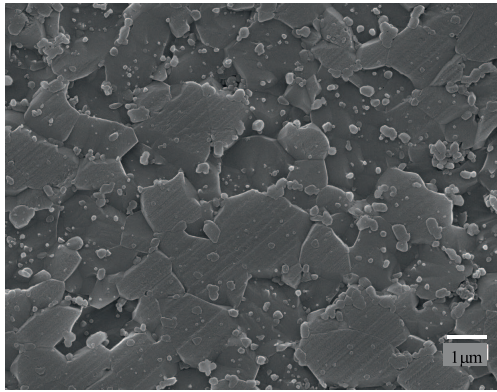


Fig. 1. Microstructure of the nanocomposite containing 10 vol% SiC

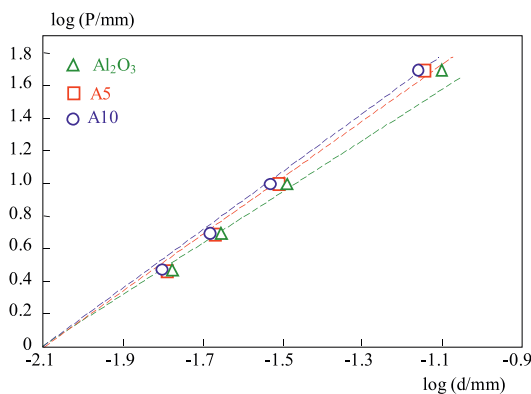


Fig. 2. Dependency of log *P* on log *d* according to Meyer law for tested materials

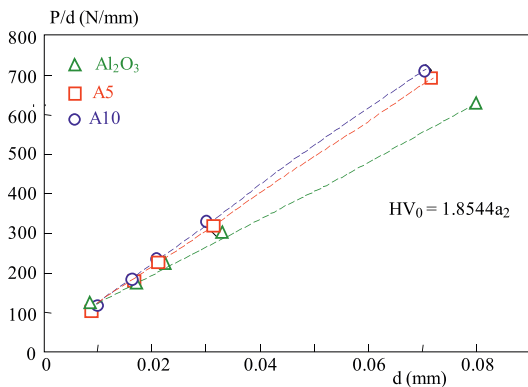


Fig. 3. Dependency of *P/d* on *d* according to the PSR model for tested materials

Table 1. Vickers hardness of the studied materials

Sample	HV5 (GPa)	HV1 (GPa)	HV0.5 (GPa)	HV0.3 (GPa)	HV0.1 (GPa)
Al ₂ O ₃	14.3 ± 1.0	17.1 ± 0.8	17.9 ± 1.1	19.3 ± 0.6	21.3 ± 1.7
A5	17.9 ± 0.4	19.5 ± 0.5	20.4 ± 1.0	20.5 ± 0.6	20.6 ± 0.5
A10	18.7 ± 0.3	20.8 ± 0.3	20.9 ± 0.4	21.2 ± 0.5	21.7 ± 0.5

Generally, nanosized SiC particles hindered the grain growth of Al₂O₃. The sample Al₂O₃ + 10 vol% SiC consisted of fine microstructure. A small number of processing flaws in the form of pores or clusters of SiC grains could be identified. Location of the *intra*- and *inter*-granular inclusions within Al₂O₃ matrix is shown in Fig. 1.

The macro- and micro-hardness of Al₂O₃/SiC composites is shown in Tab. 1. According to the results for all investigated materials the hardness increased with decreasing indentation load and with increasing volume fraction of SiC additive. The lowest hardness was found for the alumina but with decreasing load its hardness increased faster in comparison with those of the composites and at the lowest indentation load the hardness values of all materials were very similar.

Figure 2. illustrates the Meyer law parameters determined by the regression analyses of the results. According to the results the most significant ISE was found in alumina (*n* = 1.83) and the ISE observed in the composites (*n* = 1.92 and *n* = 1.93) was much less pronounced. These values lie within the range for *n* of 1.748 to 1.979 obtained for a variety of ceramics and glasses with indentation loads from 5 to 50 N by Gong *et al* [9]. Like Gong *et al*, we found radial cracking at the corners of the indents for all tested materials in the whole range of applied loads. Gong *et al* pointed out that this may affect the hardness values obtained but since it is difficult to suppress this cracking, the extent of its influence on hardness is not clear.

The evidence here is that the nanocomposites showed smaller scatter in hardness than the pure alumina which indicates that inhomogeneities in particle distribution do not significantly affect the hardness. The better defined hardness in the nanocomposites may be a consequence of the suppression of surface microcracking in these materials by the SiC particles within the alumina grains [13, 14].

Figure 3. shows the *P/d*-*d* curves for the tested materials. True hardness was calculated for each material. According to Li and Bradt [2] who investigated the microhardness indentation load size effect in TiO₂ and SnO₂ single crystals, if the fact that the power-law exponent, *n* < 2 is the result of not taking the proportional specimen resistance of the test specimen into account, then there must exist an inverse correlation between *n* and the *a*₁ values that describe the proportional specimen resistance (PSR) model. This shows that both Meyer law and the PSR model give reasonable mathematical fits to data exhibiting an ISE but there is nothing in this analysis supporting any particular physical interpretation.

The values for *a*₁ shown in Fig. 3 are significantly smaller for the nanocomposites (*a*₁ = 28.1 N/mm, *a*₁ = 29.4 N/mm) than for the alumina (*a*₁ = 53.6 N/mm), indicating according to the physical rationalisation of the PSR model a lower “specimen resistance” to indentation in the nanocomposites. One reason for this may be that the large, deviatoric thermal residual stresses in the nanocomposites help to initiate plastic deformation under low indentation loads. Using the Selsing formula [15] and the physical properties of the matrix (*m*) and particle (*p*), $\alpha_m = 8.8 \times 10^{-6} \text{K}^{-1}$, $E_m = 380 \text{ GPa}$, $\nu_m = 0.21$ and $\alpha_p = 4.7 \times 10^{-6} \text{K}^{-1}$, $E_p = 490 \text{ GPa}$, $\nu_p = 0.19$, the matrix residual stresses close to the particles can be calculated to be approximately $\sigma = -2 \text{ GPa}$ in the radial direction and +1 GPa in the tangential direction. Stresses

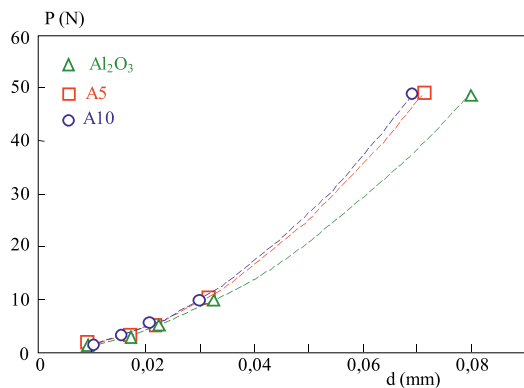


Fig. 4. Dependency of P on d for tested materials according to the MPSR model

of this magnitude have also been confirmed experimentally [16]. A further reason for the ease of initiation of plastic deformation in the nanocomposites may be that the alumina grains of the nanocomposites are observed to contain many dislocations [10] even in the as processed condition, so there is no need to nucleate new dislocations in the early stages of indentation.

The term a_2 from the linear fits in Fig. 3. describes the load independent, so called “true hardness”, which was found for Al_2O_3 , A5 and A10 to be 13.2 GPa, 17.3 GPa and 18.0 GPa, respectively.

Gong et al [9] investigated the ISE in ceramics with fracture toughness from $0.8 \text{ Mpa}\cdot\text{m}^{0.5}$ to $12.4 \text{ Mpa}\cdot\text{m}^{0.5}$. They found that for some ceramics the PSR model does not provide a satisfactory explanation of the ISE and offered a modified PSR model to solve this problem, see equation 4. The term P_0 in this model was rationalised by Gong *et al* in relation to the residual surface stresses in the test specimen associated with the machining and polishing of the samples prior to testing.

In Fig. 4. the relationship between P and the indentation size d is illustrated in the form of polynomial curves with the calculated parameters of the modified PSR model. The values for a_1 are for the nanocomposites A5 and A10 ($a_1 = 65.4 \text{ N/mm}$, $a_1 = 81.7 \text{ N/mm}$) respectively, for the alumina $a_1 = 108.2 \text{ N/mm}$. The correlation is very good, although the introduction of an extra adjustable parameter (P_0) is bound to lead to improved fitting, whatever the correct physical explanation of the ISE. In the present case, the values of P_0 were negative for the monolithic alumina and for the composites too. There is therefore no systematic trend occurred which may relate to microstructure or surface residual stresses from machining.

The MPSR model results in slightly lower “true hardness” values of 11.9 GPa, 16.3 GPa and 16.7 GPa, for the Al_2O_3 , A5 and A10 ceramics, respectively, although the trend in hardness with SiC addition is the same as was found for the PSR.

4 CONCLUSION

The strongest ISE was found for alumina with a Meyer index of $n = 1.83$. The lower ISE for the nanocomposites

was attributed to the high thermal stresses and pre-existing dislocation distributions in these materials. The PSR model can be used to analyze the ISE observed in all of tested materials. According to the modified PSR model results, in comparison with Vickers hardness exhibits lower values due to cracking. No evidence was found for the influence of machining stresses on the ISE and it is likely that the introduction of P_0 in the modified PSR model improves the fit to results mainly by providing an extra adjustable variable rather corresponding to a simple physical phenomenon.

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