Oral ICF10343

PLASTIC FLOW AND FRACTURE OF BULK NANOCRYSTALLINE CERAMICS AND INTERMETALLICS IN INDENTATION

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ABSTRACT
A study in model mechanics is performed to characterize the plastic flow and fracture in indentation of the full density nanocrystalline ceramics and intermetallics. An analysis of a load-depth curve in Berkovich indentation makes it possible to derive a relatively low level of 4.4 GPa for the value of the yield stress(σ_{yn}) of the nanocrystalline ZrO_2-20mol%Al_2O_3 sample, when assuming the non-strain hardening property. The dynamical indentation verifies that the yield stress for the fully dense nanocrystalline TiAl sample follows the formula of $\sigma_{yn}=\sigma_{ya}[1+V_f(E_c/E_a-1)]$ that is constructed on the basis of the rheological model for the nanocrystal with an amorphous network volume(V_f). The fracture toughness(K_{IC}), as estimated from the length of a crack occurred at the corners of pyramidal Vickers indent, exhibits an extremely high value of 34 MPa $m^{1/2}$ for the forged sample of nanocrystalline PSZ-20mol%Al_2O_3 consisting with the cubic and tetragonal phases.
KEYWORDS
amorphous ceramics, nanocrystallization, full density consolidation, yield stress, fracture toughness,

INTRODUCTION

The fully dense nanocrystalline material is becoming one of the most promising materials that have the outstanding mechanical characteristics necessary to realize a variety of the near-future technologies[1]. The author has proposed the non-equilibrium P/M processing that consists of the mechanical alloying and the electric discharge consolidation for the synthesis of the fully dense nano-amorphous materials[2][3], and then discovered an extremely high yield strength of 3.1 GPa in compression for the nanocrystalline TiAl intermetallic[4] and the high-speed superplasticity at a relatively low temperature in nanocrystalline ZrO_2-20mol%Al_2O_3[5]. A study in the model mechanics[6] has been carried out to give the formula of the super strength pertaining to the bulk nanocrystalline alloys and intermetallics using well-defined yielding[7]. Concomitantly, the Vickers hardness testing has been used to make clear the nature of the strength and the related phenomenon as a convenient method, namely in the bulk nanocrystalline materials that exhibit a brittle behavior in a uniaxial stress state at the present. Here, the author is going to provide a quantitative way of indentation to the derivation of the yield stress and the fracture toughness for the bulk nanocrystalline ceramics and intermetallics.

EXPERIMENTAL PROCEDURE

The amorphous powders of TiAl[8], ZrO_2-20mol%Al_2O_3[9] and PSZ-20mol%Al_2O_3[10] were produced by employing the rotating-arm reaction ball milling method[11]. The instrumented electric discharge consolidation method can be used to prepare the fully dense product of the nanocrystal synthesized via crystallization of the amorphous powder[3][12][13]. X-ray diffraction is used to characterize the ball milled powder and the consolidated product. The average grain size(d) is estimated from X-ray line broadening at the half of maximum using the conventional Scherrer formula. The Berkovich indentation method equipped with a depth-sensing system(Shimazu DUH-201S) is used to evaluate the yield stress in the bulk amorphous and nanocrystalline materials. The pyramidal Vickers hardness testing is employed to derive the fracture toughness for nanocrystalline ceramics on the basis of the indentation microfracture method.

RESULTS AND DISCUSSION

Plastic flow in dynamic indentation

Figure 1 shows the Berkovich indentation depth at various holding loads for the fully dense nanocrystalline ZrO_2-20mol%Al_2O_3 product, as prepared at the consolidation temperature of 1360 K[14]. The load-Bekovich indent depth curve is smooth up to the applied load of 2 N at the maximum avoiding any cracking as shown in Figure 1. The indentation depth(d_1) at the applied load of 2 N nearly equals to the depth(d_2) after holding for 60 s in the case of the lowest loading rate of 1.4 mN s^{-1}, while the difference between d_1 and d_2 increases with increasing loading rate. Therefore, the Berkovich indentation depth(d_2) here is used as an accepted parameter. For nanocrystalline ZrO_2-20mol%Al_2O_3, the load-depth relations are almost identical at both loading rates of 1.4 and 71 mN s^{-1} as shown in Figure 1. Derive the yield stress in dynamical indentation. The formula for the load(P)-indentation depth relation has been constructed using the finite element method by Larsson et al.[15], and is applied to the quantitative evaluation of the yield stress(σ_y) at room temperature for the bulk amorphous and nanocrystalline materials as follows:

$$P=1.273(\tan 24.7^{o})^{-2}\sigma_y(1+\sigma_r/\sigma_y)(1+\ln E\tan 24.7^{o}/3\sigma_y)d_2^2 \quad (1)$$

where σ_r and E are the flow stress and Young s modulus. The analysis of the experimentally obtained load-depth relation on the basis of the equation (1) gives a relatively low level of 4.4 GPa for the value of the yield stress of the nanocrystalline ZrO_2-20mol%Al_2O_3 product with the average grain size of 11nm for a tetragonal phase, together with the proper value for Young s modulus. In this calculation, it is reasonably assumed that σ_r equals to the value of σ_y , when considering that the bulk nanocrystalline material is characterized as a nearly non-strain hardening solid as well as the amorphous alloy[7].The thus-derived yield stress can be coupled with the relatively low level of approximately 1000 DPN for Vickers hardness number(H_v) for the fully dense nanocrystalline ZrO_2-

Figure 1: The relationship between the holding load and depth in Berkovich indentation for fully dense nanocrystalline ZrO_2-20mol%Al_2O_3 sample, prepared by the electric discharge consolidation of the amorphous powder.

Figure 2: The load-depth relation in Berkovich indentation for the fully dense TiAl as consolidated below the glass temperature. The solid and broken lines are respectively the predictions for amorphous and nanocrystalline TiAl on the basis of the formula that is constructed on the basis of the rheological model. This figure includes the load-depth curve in the case of loading rate, 1.4 mN s^{-1}.

20mol%Al_2O_3 sample by a relation of $H_v/\sigma_y=\alpha$ where α is a constant ranging from 2 to 3 for amorphous and nanocrystalline alloys. Note that the fully dense nanocrystalline covalent and oxide ceramics, SiC[16] and PSZ-20mol%Al_2O_3[17] have two-third level of Vickers hardness numbers of ceramics as prepared by the conventional method. The yield stress at the room temperature is an important material parameter to discuss the mechanical characteristics such as softening, ductilization and enhanced fracture toughness in the bulk nanocrystalline ceramics as well as metals and alloys. Figure 2 shows the load-depth relation in Berkovich indentation for the fully dense amorphous and nanocrystalline TiAl samples. So far, the compressive yielding in nanocrystalline TiAl has been fairly well expressed with a Hall-Petch type formula using the well-defined yield stress in the case of $d>d_c$ where d_c is the critical grain size. Here, consider the yielding in the case of $d<d_c$. The formula of the yield stress for nanocrystalline TiAl[7][19] is constructed on the basis of the rheological model as follows: $\sigma_{yn}=\sigma_{ya}[1+V_f(E_c/E_a-1)]$

(2)where V_f is the amorphous volume. For nanocrystalline TiAl, Young s modulus(E) obeys the mixture law of $E=E_cV_f+E_a(1-V_f)$ where E_c is Young s modulus for intermetallic TiAl, and a relation of $V_f=0.185d^{0.5}+0.77$ is experimentally obtained. The samples as in Figure 2 are synthesized by the consolidation of the mechanically alloyed amorphous powder and the isothermal annealing below the glass temperature at the applied pressure of 600 MPa[1][18], in order to obtain a single amorphous phase and an average grain size below the critical value(d_c). The load-depth relation for amorphous TiAl is fitted by the predicted curve of equation (1) with the yield stress(σ_{ya}) of 0.65 GPa and Young s modulus(E_a) of 40 GPa in the literature[7]. The prediction by equation (1) can give a good fit to the experimentally obtained load-depth relation in Berkovich indentation with the average grain size of 8 nm in a range of the observation by high-resolution electron microscopy[18]. In other words, a good agreement between the prediction and the experiment validates that equation (2) is applicable to the yielding in bulk intermetallic TiAl with $d<d_c$. While the equation (2) has been, so far, confirmed by a decrease in Vickers hardness number with decreasing average grain size[19].

Fracture in Vickers hardness testing

Figure 3 shows the relationship between the applied load and the length of a crack occurred at the corners of Vickers indent for the fully dense nanocrystalline PSZ-20mol%Al_2O_3 and ZrO_2-20mol%Al_2O_3 samples, and the forged sample. Evaluate the fracture toughness(K_{IC}). For a median/radial crack, the load-crack length relation is given by the following equation.

$$P/c_0^{3/2} = K_{IC}/A(E/H_v)^n \tag{3}$$

Figure 3: The relationship between the applied load and the length of a crack in indentation microfracture method using Vickers hardness testing for the fully dense nanocrystalline PSZ-20mol%Al_2O_3 and ZrO_2-20mol%Al_2O_3 samples. This figure also includes the result of the forged PSZ-20mol%Al_2O_3 sample. where c_0 is the length of a crack, A being the constant and n is the exponent. It can be seen that the applied load is proportional to $c_0^{3/2}$ for each of nanocrystalline oxide samples. Therefore, the fracture toughness can be derived by the slope by using the equation (3) with the customarily used values of A=0.016 and n=0.5, together with the proper values of E and H_v. For fully dense nanocrystalline PSZ-20mol%Al_2O_3 consisting of cubic and tetragonal phases, the fracture toughness is higher than that of submicron sized PSZ as prepared by conventional P/M methods, and shows a further increase from 11 to 12.4 MPa $m^{1/2}$ by decreasing average grain size. On the other hand, the fracture toughness for the fully dense nanocrystalline ZrO_2-20mol%Al_2O_3 samples is derived as 8.7 MPa $m^{1/2}$ although this sample has the lowest tetragonal grain size of 11 nm. This insufficient enhancement may results from the monoclinic phase involved as a second phase and an external Fe content incorporated during milling. Moreover, it is found that the nanocrystalline PSZ-20mol%Al_2O_3 sample, subjected to the high-speed superplastic forging with the compressibility of 64 %, has an extremely high fracture toughness of 34 MPa $m^{1/2}$ at the maximum within the experiment of this study. The low yield stress as stated before and the high microfracture stress give rise to the extraordinary fracture toughness in the fully dense nanocrystalline PSZ-20mol%Al_2O_3. Especially, the latter is resulting from the pore free consolidation, the synthesis of tetragonal and cubic phases, the high quality production of the amorphous powder avoiding external Fe contamination in non-equilibrium P/M processing. A further study on the indentation microfracture method is desired to evaluate an exact fracture toughness for bulk nanocrystalline ceramics, including the inspection of eqn. (3).

CONCLUSIONS

This article provides a study in model mechanics for the plastic flow and fracture in indentation of the bulk nanocrystalline ceramics and intermetallics. The Berkovich indentation is successfully used to derive a relatively low yield stress of 4.4 GPa that is coupled with the softening in Vickers hardness for the fully dense ZrO_2-20mol%Al_2O_3 sample with the grain size of 11 nm. Then, the dynamical indentation validates that the fully dense sample of nanocrystalline TiAl with $d<d_c$ follows the formula of $\sigma_{yn}=\sigma_{ya}[1+V_f(E_c/E_a-1)]$ that is constructed on the basis of the rheological model. On the other hand, the forged sample of nanocrystalline PSZ-20mol%Al_2O_3 with the cubic and tetragonal phases is found to exhibit an extremely high level of 34 MPa $m^{1/2}$ for the value of the fracture toughness, as estimated from the indentation microfracture method.

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