Close-Out Report for Grant Transfer

Grant No.: DE-FG02-05ER46195

Project Title: Size Effect in Cleavage Cracking in Polycrystalline Thin Films

PI: Yu Qiao

Reason for Closure of the Project: the PI moved to The University of California, San Diego. The project is transferred to the new institution. The research will continue at the new location. A complete technical report will be provided when the entire program is completed.

Program Scope

The reliability of polycrystalline thin films is essential to assuring safe performance of micro/nano-electromechanical systems. Usually, they are of through-thickness grain structures and are brittle at working temperatures, and therefore their fracture properties are dominated by the resistances offered by grain boundaries to cleavage cracking [1,2]. As a cleavage crack front propagates across a high-angle grain boundary, it would first penetrate across a number of break-through points [3], and the persistent grain boundary areas would then be separated through shear fracture or ligament bending [4,5]. It is, therefore, envisioned that as the film thickness is smaller than the characteristic distance between the break-through points, which is often in the range of 0.5-5 microns, the crack front transmission can be significantly confined by the film surfaces, leading to an either beneficial or detrimental size effect. That is, the fracture toughness of the polycrystalline thin film is not a material constant; rather, it highly depends on the film thickness. Since this important phenomenon has not received the necessary attention, we propose to carry out a systematic study on fracture resistances of bicrystal silicon films. The film thickness will range from 1 to 1000 microns, and the crystallographic orientations across the grain boundaries will be controlled precisely so that the size effect and the geometrical factors can be analyzed separately. The study will starts with thick films. Once the crack front transmission process is relatively well understood, it will be extended to thin films. This project will shed light on crack-boundary interactions in confining microenvironments, which has both great scientific interest and immense technological importance to the development of fine-structured devices.

Recent Progress

The testing samples of thicknesses in the range of 100 μm to 1 mm have been successfully prepared, as shown in Fig.1(a), for which we have developed lab techniques of combined polycrystal characterization and thermal modification as well as bicrystal harvesting and thinning to overcome the following hurdles: (1) the sample must contain and only contain two large grains with the crystallographic orientations being significantly different, i.e. the grain boundary must be high-angle; (2) the grain boundary must be clean and oxide free; (3) the samples thickness should be controllable in a broad range so as to investigate the size effect; and (4) most importantly, a pre-crack must be produced with the crack tip arrested by the grain boundary so that the fracture resistance of the grain boundary can be measured.

Preliminary fracture experiment on these silicon bicrystal samples is being carried out. The results are quite encouraging. As shown in Fig.1(b), it is clear that the fracture surface shifts form the cleavage plane of grain “A” to that of grain “B” across the grain boundary. Different from what we observed in iron-silicon bicrystals [6], the front transmission in a silicon sample does not demand shear deformation or fracture of the grain boundary itself; rather, separation of a secondary cleavage plane in grain “A” takes place, by which the crack can “channel” through the boundary affected zone. Detailed fractography study is in progress to understand the details of this phenomenon.

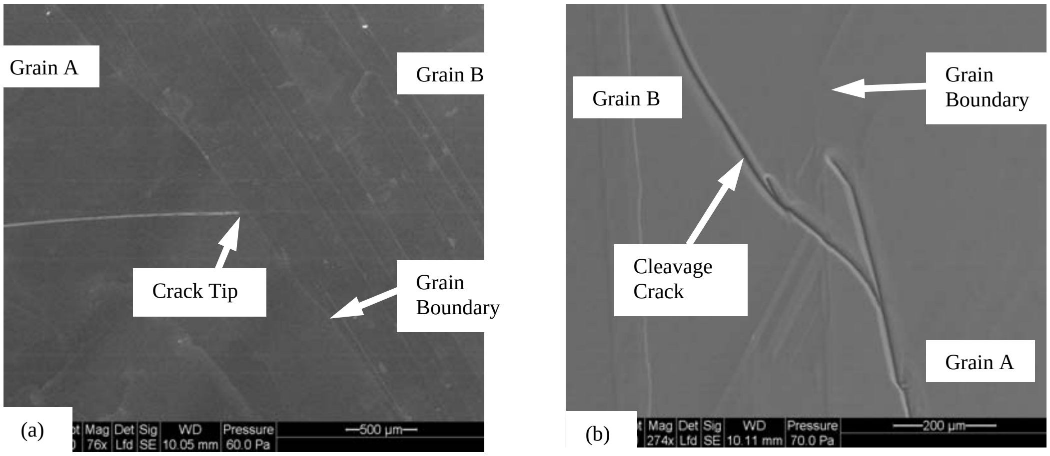

Fig.1 SEM microscopy of (a) a cleavage crack tip arrested by the high-angle grain boundary of a boron doped silicon bicrystal and (b) a cleavage crack across the high-angle grain boundary of a boron doped silicon bicrystal.

Based on the analysis of energy balance of cleavage front propagation in heterogeneous environments, the upper limit and lower limit of the size dependence of grain boundary toughness have been predicted. The upper limit is reached when persistent grain boundary areas sufficiently tough such that the final separation of crack flanks occurs after the crack trapping effect is fully overcome (see Fig.2). Under this condition, there transition range of film thickness around 1-2 times of the width of break-through windows in which size effect is pronounced. In the transition range, as the film thickness increases, the grain boundary toughness becomes larger. Below or above this range, the grain boundary toughness is somewhat size independent. The lower limit is obtained as the persistent grain boundary areas fail during the crack front transmission process. In this case, the film thickness effect is pronounced for cracks shorter than 500 times of the film thickness.

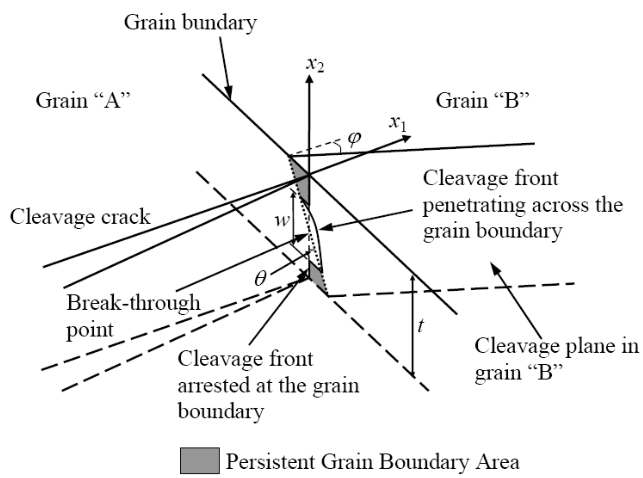

Fig.2 A schematic diagram of the cleavage front transmission across a high-angle grain boundary.

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Future Work Plans

We plan to continue the experimental study on silicon thin films of various thicknesses. For relatively thick films of thicknesses larger than 100 μm, the sample preparation techniques have been well established and the investigation will be focused on the dependence of grain boundary toughness on crystallographic misorientations. The precracked bicrystal samples will be fractured under nearly pure mode-I loadings. Due to the large number of available cleavage planes in silicon and the large T-stress, the misorientation angles should be smaller than 20^o. The development of break-through points and the behaviors of persistent grain boundary areas will be observed directly on the fracture surfaces, which will provide an important basis for determining the thickness of silicon thin film samples.

Silicon thin film samples will be fabricated from the thick film samples via chemical-mechanical polishing and enhanced etching. The sample thickness will be controlled from less than 1 μm to 100 μm. The grain boundary toughness will be measured under both mode I and mode II loading. The former is relatively simple and suitable for theoretical analysis; the latter is more close to the working conditions of micro/nano-electromechanical system components. Since the final thickness is quite small, a protection frame will be produced surrounding the sample during the polishing and etching processes, and be broken off just before the fracture test. All the residual stresses in the thin film caused by the deposition, polishing, and etching will be released when the protection frame is broken. If necessary, high-temperature annealing will be performed to further lower the level of residual stresses. Based on the experimental data, as well as the SEM and TEM analyses, the factors governing the boundary toughness can be discussed quantitatively. If possible, an environmental chamber will be designed to perform the microscale fracture experiment at temperatures in the range of 20-600^{o}C, which will shed light on the influences of the dislocation behavior and the grain boundary shearing. A number of promising experiments will be analyzed in detail to relate the plastic deformation to their slip ingredients by a variety of available methods such as etch pitting, polarized optical microscope observation, and electron back scatter diffraction. Note that the influence of free surfaces (or film thickness) and adjacent grains (or grain size) can be quite different, which can be examined by testing free-standing and rigid-substrate-supported samples of similar grain structures.

The measured boundary toughness can be used in a variety of methods to estimate the global cracking resistance of fine-grained materials. Here, different modes of possible crack advance must be considered, ranging between monolithic quasi-straight crack fronts to intricately percolating cleavage cracking processes through grains as conceived by McClintock [2]. To establish the dominant form of crack advance, fracture experiments in textured and non-textured polysilicon samples with various grain sizes will be performed, followed by examining the cleavage river markings through the field of fractured fine grains. The simplest method to assess the global fracture resistance could be a variant of crack trapping models that will amount to a crack-front-line-average of the individual energy release rate of neighboring grains as in the model of Rose [7], which has been found to be reasonably accurate when compared with the more elaborate crack trapping model of Bower and Ortiz [8], as was verified by Mower and Argon [9]. If the advance of the macrocrack is found to be more tortuously percolating through grains as noted by Becham and Pellox [10] and considered by McClintock [2], a more detailed numerical simulation will be necessary. The actual result is likely to be in between the monolithic and the fully tortuous cases, where the former is likely to result in a position independent cracking resistance while the latter may have the elements of a rising resistance, i.e. R-curve, form. The important size effect on the global fracture resistance can then be discussed by incorporating the model of the resistance of individual grain boundaries into this framework. In order to gain a deeper insight into the crack front transmission behaviors, especially for thin films of thicknesses less than 1 μm, a molecular dynamics enhanced finite element analysis will be conducted to visually demonstrate the effectiveness of the important factors, such as film thickness, crystallographic misorientations, as well as grain boundary structures.

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Y. Qiao, X. Kong, "On size effect of cleavage cracking in polycrystalline thin films," *Mech. Mater.*, to be published.
Y. Qiao, X. Kong, "Cleavage resistance of fine-structured materials," *Met. Mater. Inter.*, to be published.
Y. Qiao, X. Kong, S. S. Chakravarthula, "Energy equilibrium of cleavage front transmission across a high-angle grain boundary in a free-standing silicon thin film," *Eng. Fract. Mech.*, to be published.