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Final Technical Report
1 October 2003 – 15 January 2008

G.A Rozgonyi and K.Youssef
*North Carolina State University
Raleigh, North Carolina*

Subcontract Report
NREL/SR-520-44375
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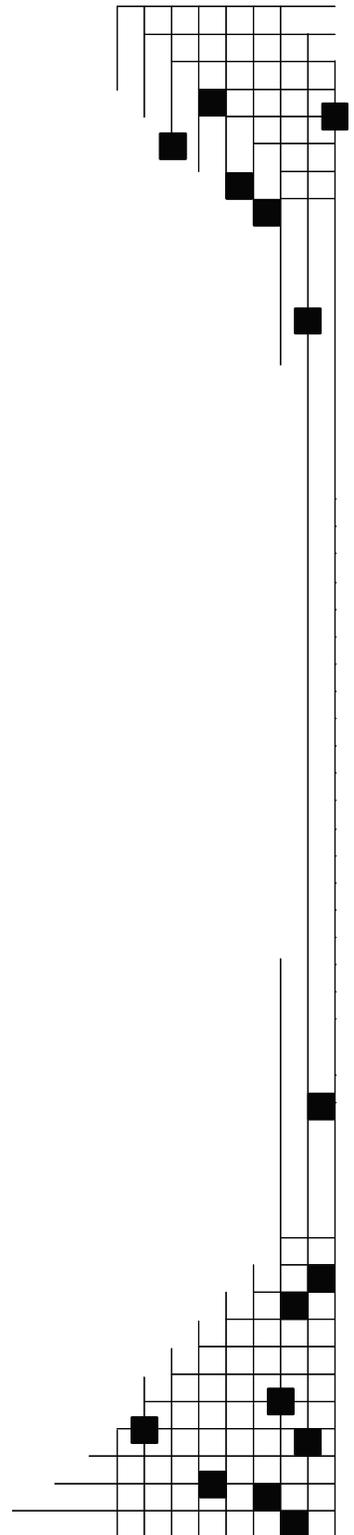
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NREL Technical Monitor: Fannie Posey Eddy
Prepared under Subcontract No. AAT-2-31605-05

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National Renewable Energy Laboratory
1617 Cole Boulevard, Golden, Colorado 80401-3393
303-275-3000 • www.nrel.gov

NREL is a national laboratory of the U.S. Department of Energy
Office of Energy Efficiency and Renewable Energy
Operated by the Alliance for Sustainable Energy, LLC

Contract No. DE-AC36-08-GO28308

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This publication received minimal editorial review at NREL



1. Introduction

Light element impurities, (e.g., O, C, N) and structural defects (dislocations, twins, and grain boundaries) are known to influence the mechanical properties of mono and multi-crystalline silicon. For example, Yonenaga and Sumino [1] showed that yield stress in silicon is a function of both the initial dislocation density and the interstitial oxygen content. It has also been found that in silicon containing point defects, dislocations, and planar defects, either the influence of oxygen on the movement of dislocation, or the blocking of dislocations by planar defects, such as grain boundaries or twin boundaries, may control the mechanical behavior [2, 3].

Since the above-mentioned impurities and defects are linked directly to the minority-carrier lifetime in silicon and therefore to its efficiency as a solar cell, we investigated in this study the mechanical properties of polycrystalline silicon in order to determine if the conversion efficiency is related to easily measurable mechanical properties. Accordingly, the dependence of hardness, elastic modulus, and fracture toughness on the wafer minority-carrier lifetime using a nanoindentation technique has been investigated in this work.

2. Materials and Methods

A cast 5" x 5" polycrystalline silicon wafer (175 μm thick) produced by BP Solar was tested. This wafer was cut from a region near a corner of the quartz crucible. Minority-carrier lifetime mapping was performed on a 7% HF passivated wafer using an AMECON JANUS 300 microwave photoconductive decay (μPCD) system.

Nanoindentations were made on selected low- and high-lifetime areas using a Hysitron TriboIndenter. Two different indenter tips were employed having centerline-to-face angles of 45° (cube corner) and 65.3° (Berkovich). Loads were varied from 0.25 to 9 mN and loading/unloading rates were kept at 1 mN/s. Five measurements were made for each load in order to check

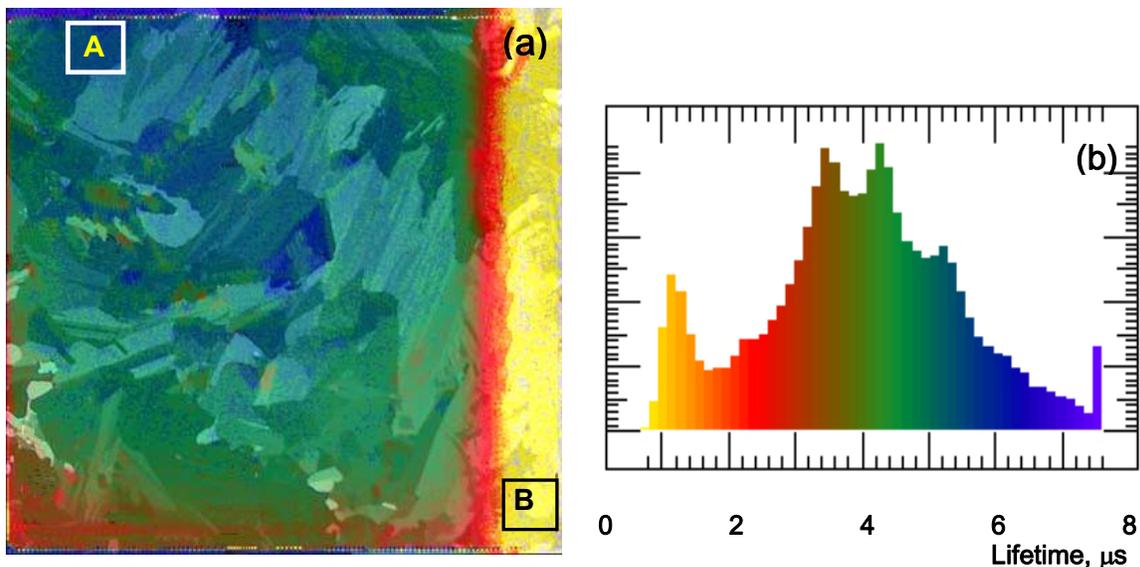


Fig. 1. A μPCD map of the polycrystalline Si wafer (a) with the corresponding lifetime histogram (b).

reproducibility of the data. After testing, all the hardness impressions were imaged using atomic force microscopy (a unit attached to the Hysitron TriboIndenter) and a JEOL 6400F field emission scanning electron microscope (FESEM) to determine the sizes of the contact impressions and the lengths of the radial cracks. Crack length and indent size were measured from FESEM images approximately 24 h after indentation, and the average values were calculated for each load.

3. Results and Discussion

Figures 1a and b are the μ PCD map and corresponding lifetime histogram, respectively. The microstructure of the polysilicon wafer was overlaid onto the μ PCD map after scanning the wafer to correlate the microstructure with the lifetime map (see Fig. 1a). Low-lifetime bands observed in Fig. 1a at the bottom and right-hand edges of the wafer correlate with ingot edges attached to the quartz crucible. The low-lifetime bands are likely due to impurities diffusing from the crucible wall. Selected high- and low-lifetime regions A and B, (see Fig. 1a) were cut from the wafer and polished for nanoindentation analyses.

Figure 2 shows typical nanoindentation load- displacement ($P-h$) curves of the low- and high-lifetime regions at three different peak loads using a Berkovich indenter. A unique material behavior called “elbow” occurs in all curves during the unloading period. It has been shown to be associated with the formation

of localized amorphous silicon regions [4]. Another important feature in Fig. 2 is the similar loading slope of the curves for each region as the indentation load is increased, while a significant variation is evident between Regions A and B. This behavior indicates that different mechanical properties exist between Regions A and B. Figures 3 and 4 show the measured hardness and elastic modulus of the low- and high-lifetime regions as a function of the indentation load, respectively.

The low-lifetime region has higher hardness (~15% more) than that of the high-lifetime region as a function of the peak load. The elastic modulus of the low-lifetime region also shows ~17% higher value than that of the high-lifetime region. This is consistent with the results reported for Czochralski-grown silicon, which show increased mechanical strength with increasing oxygen and nitrogen concentrations [5]. Even though the increased hardness of the low-lifetime region can be attributed to the higher level of

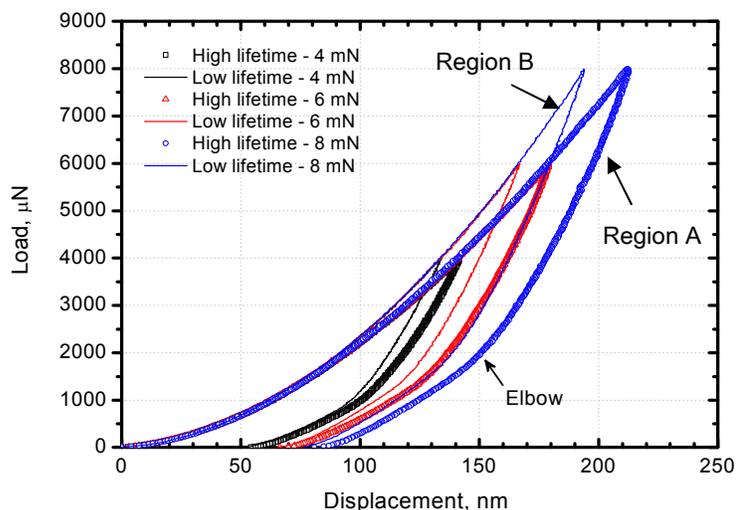


Fig. 2. Nanoindentation P-h curves in the low- and high-lifetime regions at different peak loads using a Berkovich indenter.

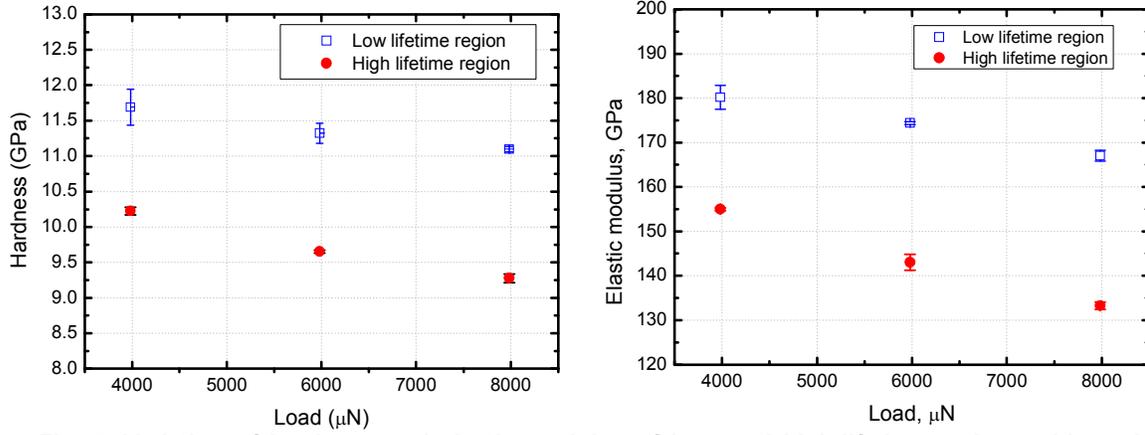


Fig. 3. Variation of hardness and elastic modulus of low- and high-lifetime regions with peak loads

impurities and the possible higher density of dislocations than found in the high-lifetime region, the variation of the elastic modulus values needs further work to be adequately explained. One possible reason could be the different crystallographic orientation between the two regions. Ebrahimi and Kalwani [6] reported elastic modulus variations in silicon from 130 to 169 GPa when the crystallographic orientation changed from (001) to (110).

Comparing the P - h curves obtained by Berkovich and cube-corner indenters for regions A and B (Fig. 4) shows that the sharper cube-corner indenter produces larger peak-load displacements and a greater proportion of permanent plastic deformation after unloading than the Berkovich indenter. The cube-corner indenter also produces load-displacement discontinuities during unloading called ‘pop-outs’, which correspond to the formation of metastable Si-XII/Si-III crystalline phases [7].

Nanoindentation impressions produced by cube-corner indenter were imaged as a function of indentation load using FESEM (images are not shown). Radial cracks were measured from these images and the fracture toughness (K_c) in the low- and high-lifetime regions was calculated using the following equation [8]:

$$K_c = \alpha \left(\frac{E}{H} \right)^{1/2} \left(\frac{P}{c^{3/2}} \right)$$

where E is the elastic modulus, H is the hardness, P is the applied load, c is the length of the radial cracks, and α is an empirical constant that was taken as 0.032 for a cube-corner tip. Figure 5 summarizes the experimental results of the fracture toughness of low- and high-lifetime regions at different loads. As can be seen in Fig. 5, the fracture toughness of Regions A and B appears to be independent of the load applied during nanoindentation, which demonstrates the applicability of the nanoindentation technique to determine fracture toughness. The average fracture toughness values of the low- and high-lifetime regions are 0.93 ± 0.04 and 0.65 ± 0.03 MPa m^{0.5}, respectively. The fracture toughness follows a trend similar to that of the hardness and elastic modulus of the low- and high-lifetime regions.

4. Conclusion

Hardness, elastic modulus, and fracture toughness of low and high carrier lifetime regions in polycrystalline silicon were evaluated using nanoindentation technique. The results obtained in this study indicate that mechanical properties of polysilicon are highly influenced by both the impurity levels and dislocation density and therefore can be correlated directly with the minority-carrier lifetime. To better understand the observed variation in mechanical properties, grain-orientation measurements (e.g., back-reflection Laue method) along with a detailed imaging technique(s) to reveal the various defects would be necessary.

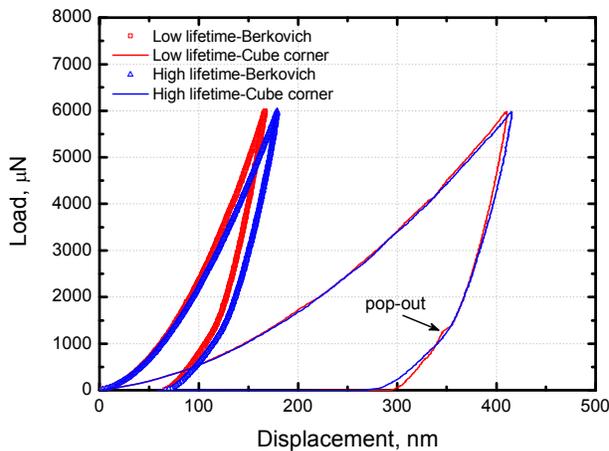


Fig. 4. Comparison of the P-h curves in the low- and high-lifetime regions using Berkovich and cube-corner indenters.

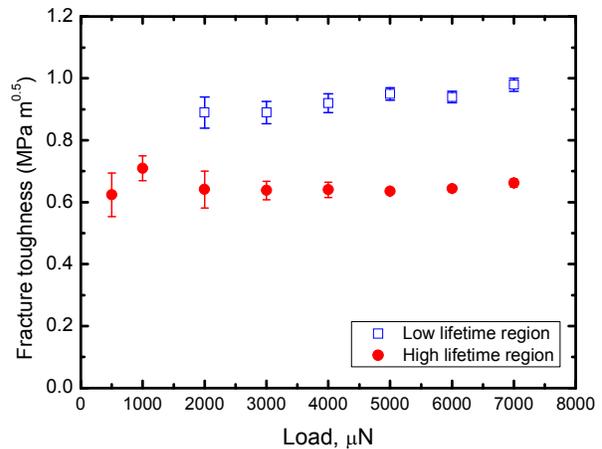


Fig. 5. Fracture toughness of the low- and high-lifetime regions as a function of indentation load.

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1. REPORT DATE (DD-MM-YYYY) November 2008		2. REPORT TYPE Subcontract Report		3. DATES COVERED (From - To) 1 October 2003 - 15 January 2008	
4. TITLE AND SUBTITLE Crystal Growth and Wafer Processing for High Yield and High Efficiency Solar Cells: Final Technical Report, 1 October 2003 - 15 January 2008			5a. CONTRACT NUMBER DE-AC36-08-GO28308		
			5b. GRANT NUMBER		
			5c. PROGRAM ELEMENT NUMBER		
6. AUTHOR(S) G.A. Rozgonyi and K. Youssef			5d. PROJECT NUMBER NREL/SR-520-44375		
			5e. TASK NUMBER PVA72501		
			5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) North Carolina State University Dept. of Materials Science and Engineering Raleigh, North Carolina 27695-7907				8. PERFORMING ORGANIZATION REPORT NUMBER AAT-2-31605-05	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Renewable Energy Laboratory 1617 Cole Blvd. Golden, CO 80401-3393				10. SPONSOR/MONITOR'S ACRONYM(S) NREL	
				11. SPONSORING/MONITORING AGENCY REPORT NUMBER NREL/SR-520-44375	
12. DISTRIBUTION AVAILABILITY STATEMENT National Technical Information Service U.S. Department of Commerce 5285 Port Royal Road Springfield, VA 22161					
13. SUPPLEMENTARY NOTES NREL Technical Monitor: Fannie Posey Eddy					
14. ABSTRACT (Maximum 200 Words) Light element impurities, (e.g., O, C, N) and structural defects (dislocations, twins, and grain boundaries) are known to influence the mechanical properties of mono- and multicrystalline silicon. Yield stress in silicon is known to be a function of both the initial dislocation density and the interstitial oxygen content. It has also been found that in silicon containing point defects, dislocations, and planar defects, either the influence of oxygen on the movement of dislocation, or the blocking of dislocations by planar defects, such as grain boundaries or twin boundaries, may control the mechanical behavior. The above-mentioned impurities and defects are linked directly to the minority-carrier lifetime in silicon, and therefore, to its efficiency as a solar cell. Therefore, in this study we investigated the mechanical properties of polycrystalline silicon to determine if the conversion efficiency is related to easily measurable mechanical properties. Accordingly, the dependence of hardness, elastic modulus, and fracture toughness on the wafer minority-carrier lifetime using a nanoindentation technique was investigated.					
15. SUBJECT TERMS PV; crystal growth; wafer process; high yield; high efficiency; solar cells; grain boundary; light element impurities; nanoindentation; polycrystalline silicon;					
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT UL	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
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