Figure 3 was purposely drawn in the form $1/t_p$ *vs.* $D$ to directly portray the light shape function $I(D)$. This plot is in contrast to the more normal plot of $D$ *vs.* $t_p$ which displays the growth of dot diameter $D$ with increasing mask-exposure time.

We consider finally the case where the mask-exposure time $t_p$ is held constant while the nonpatterned hardening time $t_n$ is varied. Equation [3] is then usefully written in the form

$$I(D) = \frac{\tau_{po}}{t_p}\left(1 - \frac{t_n}{\tau_n}\right) \quad [7]$$

We assume that $\tau_{po}$, $\tau_n$, and the shape function $I(D)$ are known, $t_p$ is given, and $t_n$ is varied. As $t_n$ is varied, the right-hand side of Eq. [7] gives the numerical value of $I(D)$, and, from the known shape-function $I$ *vs.* $D$, the expected dot diameter $D$ can then be read off.

Figure 4 shows examples of measured (data points) and calculated (smooth curves) dot diameters $D$ *vs.* the nonpatterned hardening time $t_n$. The exposure mask was the same dot mask used for the Fig. 3 measurements, and the shape function $I(D)$ used in the calculations is that shown in Fig. 3. All data in Fig. 4 were taken on two coatings. One was mask-exposed with Hg 365 mμ light with $t_p = 0.7$ min, the other with $t_p = 1.4$ min. The various nonpatterned hardening times $t_n$ were obtained by superimposing on the dot exposures flood exposures with Hg 436 mμ light in a large-area checkerboard pattern. The flood-exposure times in the various checkerboard squares provided the various $t_n$ abscissa values in the figure.

As $t_n \rightarrow \tau_n$, the developed dot diameter increases catastrophically, and the coating becomes undevelopable. This region (to the upper right of the dashed curve in Fig. 4) is readily recognized in practice and avoided. In the region to the lower left of the dashed line, the developed dots look innocently as if no nonpatterned hardening occurred. However, the coating photosensitivity to mask exposures would be seriously overestimated if the underlying nonpatterned hardening were ignored. In practice, underlying nonpatterned hardenings come from flood exposures and from coating dark hardenings.

Since the catastrophic increase of dot diameter with $t_n$ is so sensitive to $\tau_n$, the two $\tau_n$ values in Fig. 4 (4 and 4.5 min) were separately chosen empirically for best fit of the theory to the data points.

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# Damage-Free Polishing of Polycrystalline Silicon

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Recently there has been considerable interest in using polycrystalline silicon for solar cells. Although cells of about 10% efficiency can be fabricated on polycrystalline silicon substrates, it is expected that higher efficiencies can be obtained if some of the grain-boundary effects, which limit the cell performance, are well understood and, thereby, subject to moderation. Much of this investigative work to study photovoltaic mechanisms at grain boundaries involves electro-optical techniques. For this purpose, it is necessary to polish the surface of a polycrystalline silicon sample such that surface damage and profile variations, in particular at the grain boundaries, can be minimized. This paper reports a polishing technique which yields a damage-free polished surface with grain-to-grain surface height variation, δ, less than 0.05 μm. A method for monitoring surface damage due to polishing in order to optimize the polishing conditions is also described.

The problems associated with polishing polycrystalline silicon can be generalized as follows: (*i*) both chemical and mechanical (using fine grit) polishing result in steps at the grain boundaries due to different removal rates for various grain orientations; (*ii*) mechanical polishing results in surface damage giving rise to a high surface recombination velocity (which has deleterious effects on solar cell performance and complicates the interpretation of measurement data); (*iii*) chemical polishing/etching generally causes "grooving" at the grain boundaries due to the fact that the etch rate at grain boundaries is often higher than that of the grains. From these considerations it is clear that polishing to minimize surface damage and δ requires a sequence of mechanical polish followed by chemical/mechanical polish. The chemical/mechanical polishing should be done just long enough to remove the surface damage from mechanical polishing. We have tried several mechanical and chemical/mechanical polishing procedures and describe the one that gave best results.

## Procedure and Results

Polycrystalline silicon samples that were polished had sawn and etched surfaces. These wafers have considerable surface roughness and their grain structure is clearly visible, as is shown in Fig. 1. Various grains show up due to variations in reflectivity caused by changing grain inclination with respect to the incident light. Wafers were first lapped with alumina powder (typical grit size ~ 30 μm) using a felt pad[1] for about 30 min. The lapped sample exhibits a grayish appearance and no grains are visible under normal visual inspection. A heavy surface damage was evidenced by a lack of surface photovoltage signal due to high surface recombination velocity (1).

Samples were then polished with oil-based diamond (typical grit size ~ 2 μm) while gradually reducing pressure from 5 to 2 psi and maintained at this value for 2 min. Diamond polishing removed much of the lapping damage, there is still residual surface stress

Key words: polycrystalline, grain boundaries, surface photovoltage, Nalcoag.

[1] A laboratory-type machine with single spindle and rocking arm was used for all polishing steps.

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**Fig. 1. Photograph of a polycrystalline wafer before polishing. Size: 2.5 × 2.5 cm².**

which must be removed. This is done by a chemical/mechanical polish using Nalcoag slurry, Type 2350.[2]

To optimize polishing conditions, as discussed earlier, surface photovoltage technique has been used to monitor the degree of surface damage based on the fact that the surface voltage signal developed across the sample is a function of surface recombination velocity (1).

Reduction in surface damage by chemical/mechanical polishing was compared by polishing the samples for short intervals of time and measuring its photovoltage. Light intensity and the gap between top electrode and sample surface were held constant throughout all measurements. Variation of photo-signal at 10 sec intervals of polishing time is shown in Fig. 2. The solid circles show this signal variation. The decreased signal after 60 sec of polish was felt to be caused by insufficient flow of Nalcoag during polishing since repeating the experiment on another wafer yielded the signal value shown by the triangle. Saturation of signal occurs after about 60 sec of polishing, implying the surface to be free from damage.

[2] Nalcoag is a colloidal silica slurry manufactured by Nalco Chemical Company.

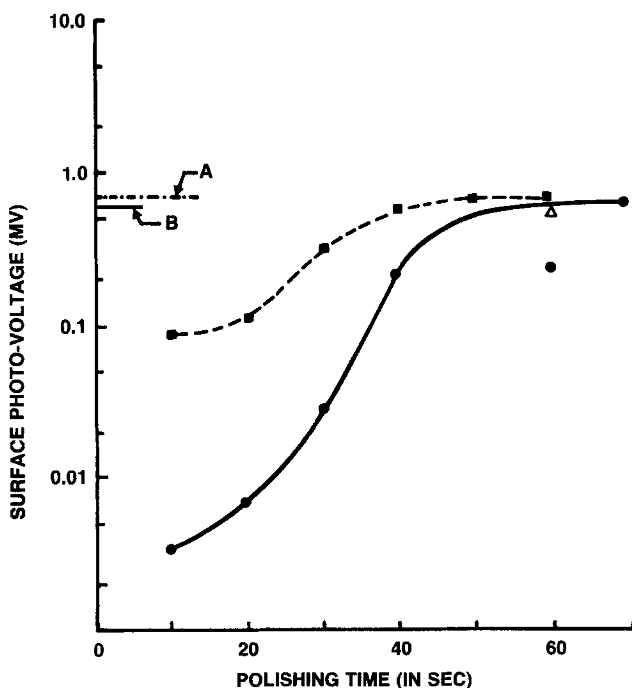


**Fig. 2. Amplitude of SPV signal vs. polishing time for Wacker Silso polycrystalline silicon. Constant light intensity and optical beam size (λ = 1.0 μm).**

In order to compare (qualitatively) the residual surface damage after lapping and diamond polishing with that of diamond polishing alone, the above sample (after Nalcoag polish) was given a diamond polish followed by repolishing with Nalcoag for 10 sec intervals and remeasuring the SPV signal. These data are shown by squares in Fig. 2. Residual damage from diamond polishing alone was much less than when the sample was lapped prior to diamond polish. Furthermore, the signal saturation occurred after only 40 sec of total polishing time. The two lines A and B in Fig. 2 indicate the signal levels obtained from two samples which were first Nalcoag polished and then etched using two different etchants. Since these samples were not mechanically polished, these signal levels correspond to damage-free surfaces lending support that the saturation signal level implies a damage-free surface.

The procedure was used to polish polycrystalline silicon wafers to a smoothness, $\delta_{max}$, of less than 0.05 μm with negligible (or no) surface damage. This is clearly seen from Fig. 3 which is a surface profile trace of a polished Wacker polycrystalline silicon wafer. A Nomarski photograph of a part of the polished surface is given in Fig. 4. Although the photographed region has many grain boundaries, only a few are visible in Fig. 4. A Dektak trace (accompanying the photograph) across the most prominent grain shows the height variation to be 0.03 μm. An interference photograph of a part of the surface, taken under sodium light (λ = 0.513 μm) is shown in Fig. 5.

The polishing procedure described above was established following several variations in mechanical and chemical/mechanical polishing sequences. Table I summarizes some essential features of these. It should be emphasized that procedures a, b, and c in Table I, lead to a steady-state grain-to-grain height variation (*i.e.*, δ does not change if the polishing is continued past

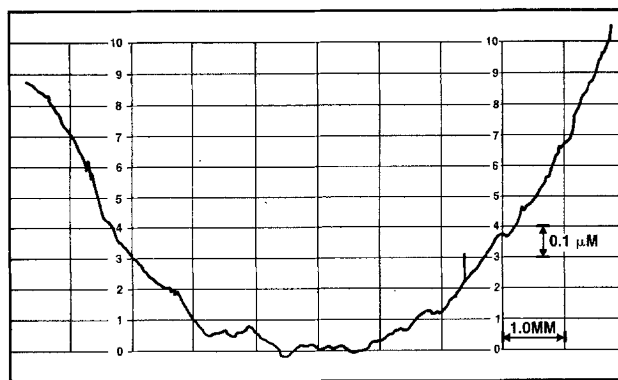


**Fig. 3. Dektak trace of the surface profile of a polished Wacker polycrystalline silicon sample.**

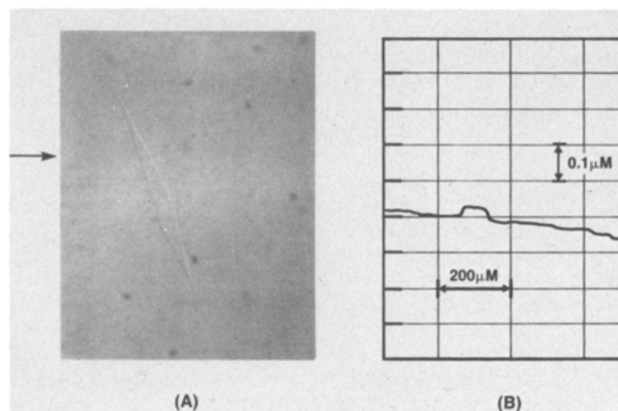


**Fig. 4. (A) Nomarski photograph of a polished wafer, magnification = 50. (B) Dektak trace across the prominent grain (along the arrow in A).**

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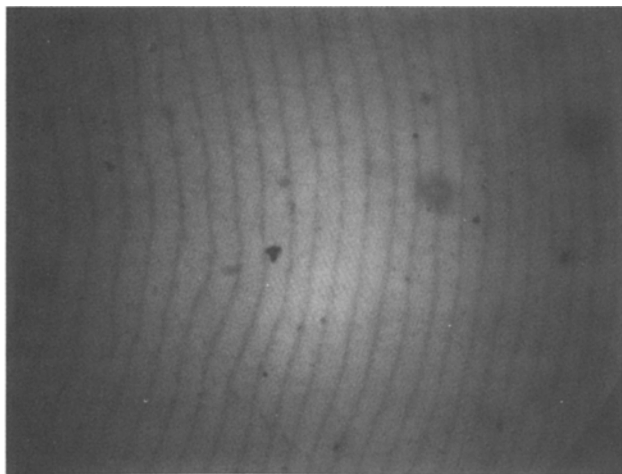


Fig. 5. Interferogram of a polished section of a polycrystalline silicon wafer.

an initial period of rapid variation). Results of chemically polishing the samples in two different etchants are also given in Table I for comparison. These etchants are often used for silicon surface preparation.

We have also found Nalcoag polishing to be a very attractive technique for wafer thinning. Figure 7 shows a plot of thickness removed *vs.* polishing time for two pressures. It is seen that removal rates are comparable to that of lapping. In addition, Nalcoag polishing allows better control for these wafers. Using Nalcoag thinning and the polishing techniques described in this paper we have been able to parallel polish RTR silicon ribbons down to a thickness of 28.1 $\mu$m for the measurement of absorption coefficients at wavelengths down to 0.7 $\mu$m (2).

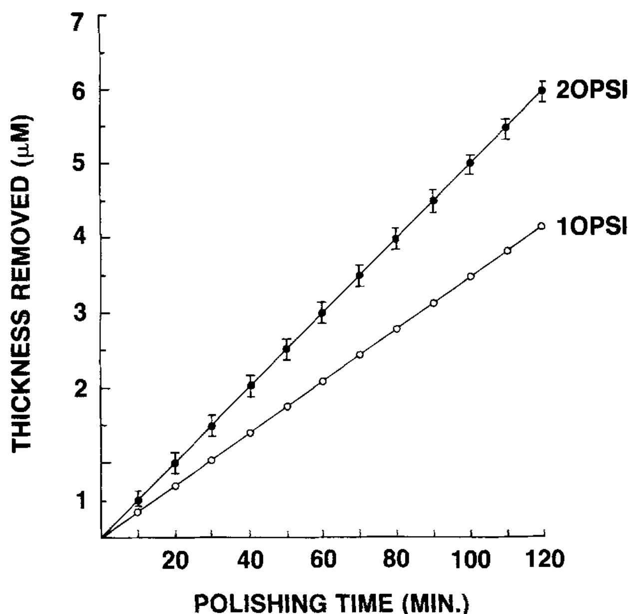
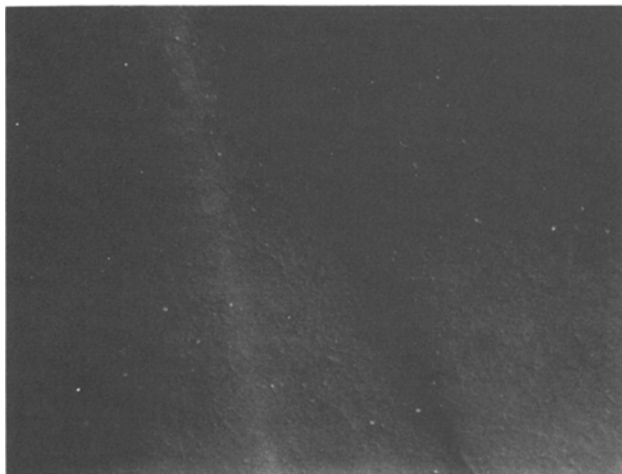



Fig. 7. Plot of thickness removed *vs.* polishing time during Nalcoag thinning.



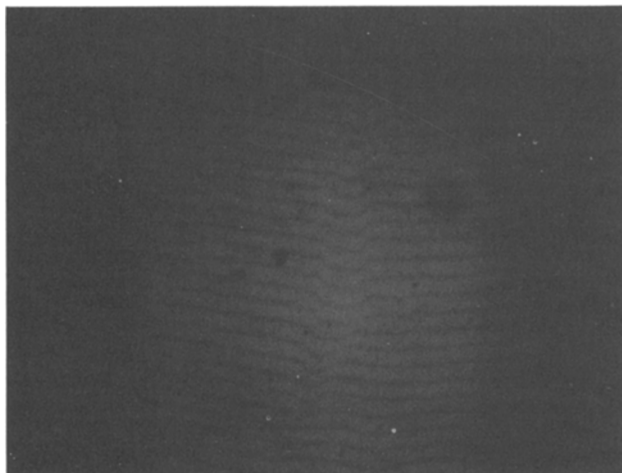


Fig. 6. (a, top) Nomarski photograph of a grain shows that damage introduced by lapping (as pits) is not completely removed by a chem mech polish for 4 min. A gentle tamer at the grain boundary is clearly seen. Magnification 400×. (b, bottom) Low magnification (10×) interference pattern of the grain in Fig. 6(a).

## Applications

The polishing of polycrystalline silicon is necessary for a variety of important electrical and optical characterization techniques. Some of these are: (*i*) focused laser scanning to determine local variations in photoresponse and to evaluate some effects of grain boundaries; (*ii*) material thinning and polishing for measurement of optical constants such as reflection and absorption coefficients, and to determine microscopic stresses by infrared birefringence; (*iii*) planar defect characterization for delineating various types of defects using chemical etchants; (*iv*) angle lapping for a variety of characterizations such as variation of defects in the bulk, junction depth measurements (at and away from grain boundaries), and a variety of other measurements (*e.g.*, potential probing) which require signal pick-off from the surface.

## Summary

The Nalcoag polishing technique for polycrystalline silicon resulted in a grain-to-grain surface height variation of $\delta < 0.05$ $\mu$m. The slope of the surface profile at grain boundaries is very gentle, and no "grooving" at grain boundaries has been observed. These properties are very important for probing grain boundaries, electrically and/or optically, along the sample surface. SPV monitoring showed the extent of surface damage to be minimal. This polishing/monitor technique has also facilitated other evaluation methods such as measurement of absorption coefficient for wavelengths near the bandedge, spectroscopic measurements of impurities like oxygen, and defect delineation by chemical etching.

## Acknowledgments

The authors wish to thank Dr. M. Coleman and Mr. J. Parsons for many fruitful discussions with them re-

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**Table I. Summary of the results of some other polishing procedures**

| Polishing method | $\delta_{max}$ ($\mu$m) | $<\delta>$ ($\mu$m) | Comments |
|---|---|---|---|
| a. Mechanical polish<br>Final grit size = 0.05 $\mu$m<br>Pad: Micro-cloth | 1.2 | 0.3 | (*i*) Grain boundaries have smooth taper<br>(*ii*) Severe surface damage indicated by very low SPV signal |
| b. Lapping + Nalcoag<br>Pressure 10 psi<br>Pressure 20 psi | <br>1.2<br>0.4 | <br>0.5<br>0.25 | (*i*) $\delta$ attains steady-state value after about 4 min of polish with Nalcoag<br>(*ii*) Lapping damage is not removed in 4 min (see Fig. 6)<br>(*iii*) Grain boundaries show gentle taper<br>(*iv*) Damage-free surface after 15 min polish with Nalcoag<br>(*v*) Useful for sample thinning |
| c. Lapping + diamond | 0.2 | 0.1 | (*i*) Leaves shallow scratches<br>(*ii*) Residual surface damage |
| d. Chemical polish<br>$HNO_3$:HF:$CH_3COOH$<br>5:1:1<br>$HNO_3$:HF:Br:$CH_3COOH$ | <br>4<br><br>4 | <br>1.5<br><br>~2 | |

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# Erratum

In the paper "Properties of Thin Polyimide Films" by L. B. Rothman which appeared on pp. 2216-2220 in the October 1980 JOURNAL, Vol. 127, No. 10 footnote one to Table I on page 2216 should read "NMP: n-methylpyrrolidinone." Also Figure 8(c) caption on page 2219 should read "after 645 hr $K = 5.12$ g/mm." In addition the last sentence before the Conclusion section on page 2220 should read "Annealing the metal at 350°C in nitrogen before test improves the Al to polyimide adhesion, resulting in a peel factor of approximately 10 g/mm after the stress cycle."

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