

PRECEDING PAGE BLANK NOT FILMED

N82 23675 D25

## FUNDAMENTAL STUDIES OF THE SOLID-PARTICLE EROSION OF SILICON\*

J. L. Routbort and R. O. Scattergood

Argonne National Laboratory

Argonne, IL 60439

## Abstract

The predictions of the theories of solid-particle erosion of brittle materials are compared to experimental results of studies in which angular  $\text{Al}_2\text{O}_3$  particles with mean diameters  $D$  of 23-270  $\mu\text{m}$  are used to erode (111) surfaces of silicon single crystals at impact angles  $\alpha$  from 20-90° and velocities  $v$  from 30-150 m/s. The description of the steady-state erosion rate by a power law,  $\dot{W} \propto (v \sin \alpha)^n D^m$  must be modified to include threshold and plasticity effects. Furthermore the velocity exponent  $n$  depends on  $D$ . Results using abrasives of different sizes mixed together can be explained using a logarithmic-normal distribution. The results of transient experiments can be used to explain the synergistic effects which are observed using a bimodal distribution of abrasives.

I. Introduction

The erosion of materials by solid-particle impacts is an important process which may limit the service lifetime of components. Brittle materials have potential uses in many high-technology energy applications, e.g. valves in coal gasification plants, gas turbine blades, electrodes and regenerative heat exchangers for MHD applications, and photovoltaic devices. Therefore, understanding the erosion process in brittle materials is important. This paper will review the progress made in the last two years in understanding the erosion process in silicon single crystals, a material which not only has applications as photovoltaic devices, but represents an ideal brittle solid and, therefore, is important as a model material that should closely conform to theoretical predictions.

II. Theory

Material removal by impacting particles occurs by lateral crack formation i.e., subsurface cracks parallel to the impacted surface, which

\*Work supported by the Basic Energy Sciences Division of the U. S. Department of Energy.

form primarily as a result of residual elastic-plastic stresses under a sharp indenter. Two models based on this experimental observation have been proposed to describe the erosion process in brittle materials. Both theories assume that the material removed is given by the area containing the lateral cracks times the depth of the lateral cracks, which is assumed to be proportional to the depth of penetration of the impacting particle. Both models assume that the lateral crack size  $c$  is proportional to the size of the radial cracks, i.e., cracks normal to the impacted surface, which form as a result of elastic-plastic loading stresses under a sharp indenter. In turn, the latter may be viewed as end-loaded half-penny cracks, the loading being due to the plastic zone expansion, where the plastic zone size is small compared to the final crack arrest size. Fracture mechanics gives  $c \propto (P_{\max}/K_c)^{2/3}$  for this situation with  $P_{\max}$  being the maximum contact force and  $K_c$  the fracture toughness. The theories differ, however, in the calculation of contact stress  $P_0 \propto P_{\max}/a_{\max}^2$ , where  $a_{\max}^2$  is the contact area.

The quasi-static model of Wiederhorn and Lawn<sup>(1)</sup> calculates the force based on the conversion of the kinetic energy of the impacting particle modelled as a sharp indenter into plastic work. On the other hand, the model of Evans, et al.<sup>(2)</sup> neglects plasticity and the contact pressure is assumed equal to the dynamic pressure when a spherical particle first hits the surface. The depth of penetration is determined from the time of contact, and the mean interface velocity, both of which are calculated from a one-dimensional impact analogue. Both models predict that the steady-state erosion rate (weight loss [g]/total weight of abrasive impacting [g]) is given by  $\Delta W \propto R^m v^n$ , where  $R$  is the particle radius and  $v$  is the velocity. The exponents predicted on the basis of the two models are given in table 1. It may be seen that the only way to distinguish between the models lies in a determination of the velocity exponent,  $n$ .

TABLE 1. Predictions of erosion models.

Model	Particle Shape	m	n
Quasi-Static <sup>(1)</sup>	Sphere	2/3 (.67)	11/6 (1.8)
	Angular	2/3 (.67)	22/9 (2.4)
Pulse-Impact <sup>(2)</sup>	Sphere	2/3 (.67)	19/6 (3.2)
	Angular	2/3 (.67)	5

### III. Experimental

Angular  $Al_2O_3$  particles were used to erode (111) Si single crystals using a slinger-type device.<sup>(3)</sup> The experimental details have been described previously.<sup>(4)</sup> Single impacts are examined using scanning electron microscopy (SEM). Erosion rates at a fixed impact angle,

ORIGINAL PAGE  
BLACK AND WHITE PHOTOGRAPH

velocity, and particle size are determined from sequential weight-loss measurements.

#### IV. Results and Discussion

##### IV-1. Single Impacts

A typical SEM of a single impact produced at  $\alpha = 90^\circ$  and  $v = 108$  m/s using  $270\text{-}\mu\text{m}$   $\text{Al}_2\text{O}_3$  is shown in Fig. 1. Each fan that originates from the impact site is formed by propagating lateral cracks which periodically diverge up to the free surface, causing material removal. The lateral crack formation is considered in detail by Evans, et al.<sup>(2)</sup> High dislocation densities under the impact sites have been observed,<sup>(5)</sup> and this provides evidence for the importance of plasticity.

Further impacts produce overlapping damage sites until eventually a steady-state  $\Delta W$  is achieved. Figure 2 illustrates a weight-loss curve measured for  $\alpha = 90^\circ$ ,  $v = 108$  m/s, using  $37\text{-}\mu\text{m}$  particles to erode a surface previously eroded into steady state using large  $270\text{-}\mu\text{m}$  particles. The erosion rate (the slope) initially decelerates as opposed to an accelerating  $\Delta W$  which is always observed on pristine surfaces. The shape of the transient is therefore determined by the initial condition of the surface.

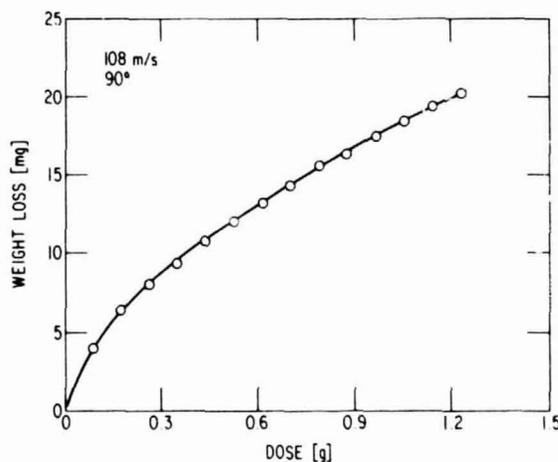
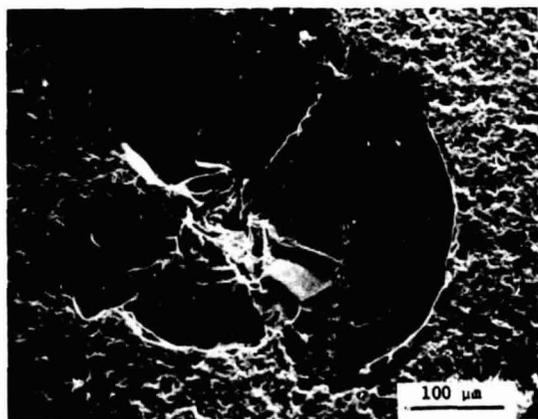


Fig. 1. (Left) SEM of single impact produced using  $270\text{-}\mu\text{m}$   $\text{Al}_2\text{O}_3$  at  $v = 108$  m/s and  $\alpha = 90^\circ$ .

Fig. 2. (Right) Weight loss as a function of dose for  $37\text{-}\mu\text{m}$  particles impacting a surface previously eroded into steady state (using  $270\text{-}\mu\text{m}$  particles) at  $v = 108$  m/s and  $\alpha = 90^\circ$ .

##### IV-2. Particle-size Dependence

The particle-size exponent  $m$  is close to the  $2/3$  predicted by the models for large particles. However, the relation greatly overpredicts  $\Delta W$

for small particle sizes. This indicates that the expression must be modified to allow for threshold effects which seem to be manifest for smaller particles. If the data for erosion rate  $\Delta W$  and particle size  $R$  of ref. 4 is plotted as  $(\ln \Delta W)/(1-R/R_0)^3$  vs  $\ln(R-R_0)^{2/3}$  to allow a comparison to the models that predict the volume removed per number of impacts, it is found that a velocity-dependent threshold size  $R_0$  can be obtained such that the relationship  $vR_0^{3/2} \approx (1280 \pm 200) \times 10^{-6} \text{ m}^2/\text{s}$  is approximately valid. For  $\alpha = 90^\circ$ ,  $v = 100 \text{ m/s}$ , the threshold  $R_0 \approx 6 \mu\text{m}$ . The threshold can be related to a critical force required to propagate a crack, and the quasi-static model<sup>(1)</sup> predicts  $vR_0^{3/2} = \text{constant}$ , while the pulse-impact model predicts  $vR_0 = \text{constant}$ . While the exact relation is difficult to evaluate, it appears from the data that the former relation is more reasonable.

#### IV-3. Particle-Size Distribution Effects

One of the difficulties which arise in the appraisal of threshold effects is that  $\Delta W$  depends on the particle-size distribution.<sup>(6)</sup> Figure 3 shows the effect of particle-size distribution at  $\alpha = 90^\circ$  and  $v = 100 \text{ m/s}$ ,

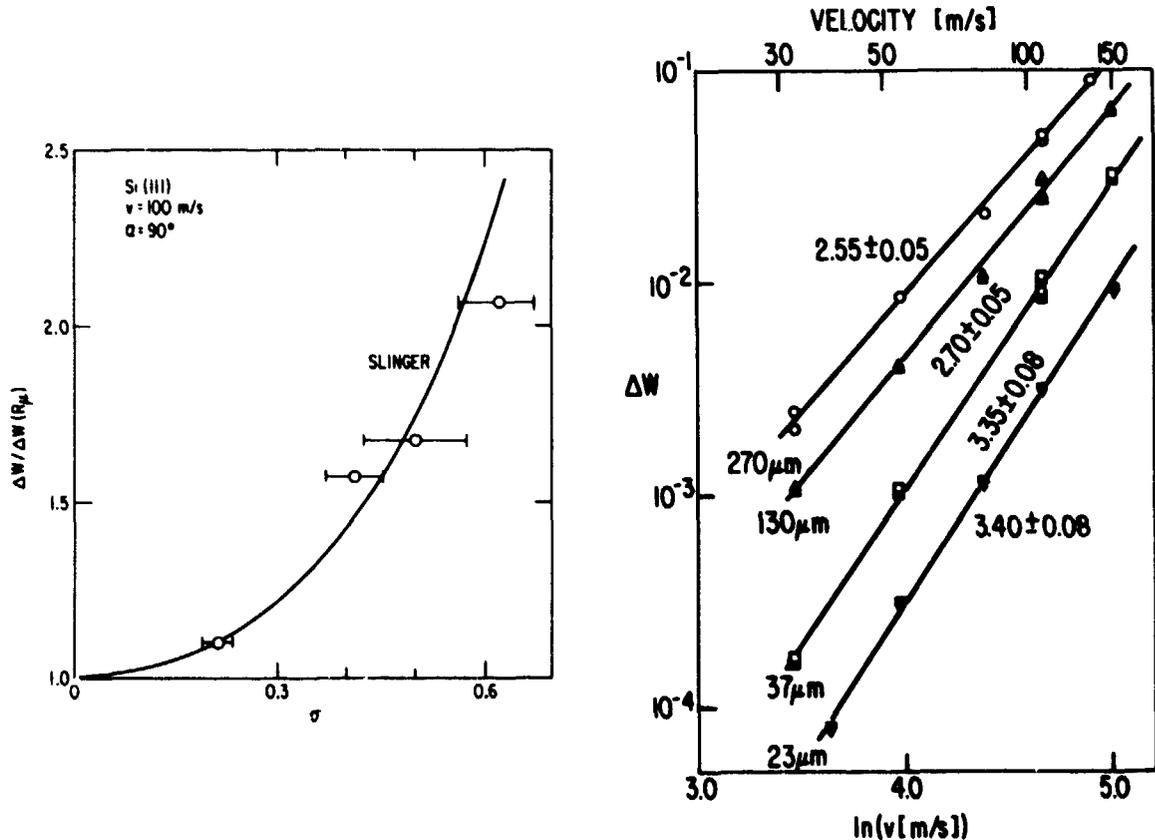


Fig. 3. (Left) The normalized erosion rate (measured steady-state rate/rate for  $\sigma = 0$ ) as a function of the particle-size distribution  $\sigma$ .

Fig. 4. (Right) The logarithm of the steady-state erosion rate as a function of the logarithm of velocity for particle sizes of 23, 37, 130, and 270- $\mu\text{m}$ .

as measured by the width of the distribution  $\sigma$ , on the normalized erosion rate (measured rate/rate for  $\sigma = 0$ ). The solid curve that is calculated from a logarithmic-normal distribution is seen to adequately describe the experimental results.

#### IV-4. Velocity Dependence

The dependence of  $\Delta W$  on  $v$  has been systematically investigated.<sup>(7)</sup> The logarithm of the steady-state erosion is plotted as a function of the logarithm of velocity for four particle sizes in Fig. 4. Good fits to  $\Delta W \propto v^n$  are obtained, but there is a dependence of the velocity exponent on the particle diameter  $D$  as shown in Fig. 5. The figure also shows data obtained on two different types of silicon carbide<sup>(8,9)</sup> which show that SiC is not unique in this respect.

The velocity exponent varies from 3.4 for 37- $\mu\text{m}$  particles to 2.55 for 270- $\mu\text{m}$  particles. As seen from Table 1, no current model can explain this variation of  $n$  with  $D$ . It may be postulated that smaller particles have shorter contact times and therefore must be approximated using the pulse-impact model, while larger particles more nearly satisfy the quasi-static model. This predicts a trend in the direction observed. It is interesting to note that the velocity exponent obtained using large (1.58-mm diameter) spheres impacting MgO is  $\approx 2.1$ ,<sup>(10)</sup> in agreement with the trend predicted in Table 1. It is believed that the exponent for hot-pressed SiC is low because of the presence of weakened grain boundaries<sup>(9)</sup> which affect the erosion rate. In this respect polycrystalline MgO behaves like MgO single crystals<sup>(11)</sup> probably because the polycrystalline MgO was relatively pure.

#### IV-5. Angular Dependence

The power-law expression for  $\Delta W$  is valid only for normal incidence for which  $\Delta W$  is maximum for a brittle solid. For oblique impact angles the velocity  $v$  can be resolved into a normal component  $v \sin \alpha$  and a tangential component  $v \cos \alpha$ . If frictionless contact conditions exist, only the normal component contributes to the erosion, and it can then be given by  $\Delta W \propto (v \sin \alpha)^n$ . Normalized data ( $\Delta W(\alpha)/\Delta W(90^\circ)$ ) obtained for various velocities and particle sizes are shown in Fig. 6, where the solid line denotes  $\sin^{2.6} \alpha$ .

The assumption that the tangential component of  $v$  does not contribute to  $\Delta W$  breaks down for  $\alpha < 45^\circ$  where the actual losses are 2-4 times greater than those predicted by the model. The additional contribution to  $\Delta W$  for smaller  $\alpha$  can be rationalized if it is assumed to be due to the tangential velocity component that arises because of a plastic-deformation cutting process, which in a ductile material has a maximum for  $\alpha \approx 20^\circ$ .<sup>(12)</sup> This is consistent with TEM observations<sup>(5)</sup> which indicates that plasticity contributes to the erosion process.

#### IV-6. Synergistic Effects

The experimental conditions used in these studies cover the range of particle sizes, velocities, and impacts generally expected in service applications where the components are subjected to an erosive environment

i.e. photovoltaic devices unprotected from a dust environment. The models, however, can not be assumed adhoc to apply to complex service conditions where, for example, several particle sizes or velocities are present simultaneously. The simplest assumption is to use a principle of linear superposition which requires that the damage processes occur independently of each other. This assumption is not in fact valid, and has recently been examined<sup>(13)</sup> in detail.

Figure 7 presents the results of an experiment designed to examine linear superposition for erosion using a mixture of two sizes of particles. The steady-state erosion rate in Fig. 7 is plotted as a function of the weight fraction of the 270- $\mu\text{m}$  particles ( $f_{270}$ ) in a mixture of 37- $\mu\text{m}$  and 270- $\mu\text{m}$  particles. The simple "law of mixing" given by  $\Delta W = f_{270} \Delta W_{270} + f_{37} \Delta W_{37}$ , where  $f_{37}$  is the weight-fraction of 37- $\mu\text{m}$  particles and the  $\Delta W$ 's are the respective steady-state erosion rates obtained for that size of particles, is shown as the dashed line and is not a valid description.

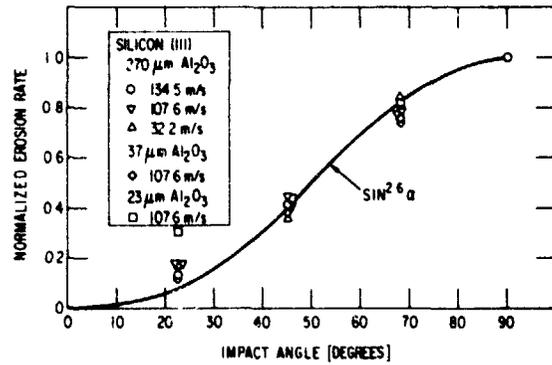
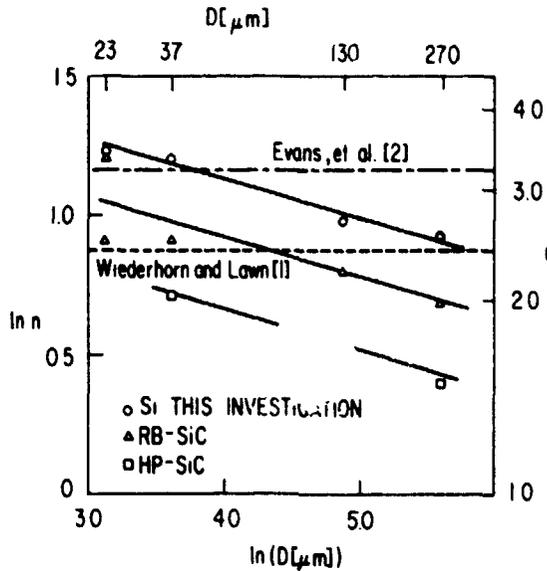


Fig. 5. (Left) The logarithm of the velocity exponent  $n$  as a function of the logarithm of the mean particle diameter  $D$ .

Fig. 6. (Right) The steady-state erosion rate normalized by  $\Delta W$  for  $\alpha = 90^\circ$  as a function of impact angle  $\alpha$ .

As can be seen from Fig. 2 the erosion rate of the 37- $\mu\text{m}$  particles impacting a surface pre-eroded with 270- $\mu\text{m}$  particles is initially enhanced over the eventual steady-state rate. The enhanced (initial) erosion rate  $\Delta W'_{37}$  may be used<sup>(13)</sup> to describe the results using  $\Delta W = f_{270} \Delta W_{270}^0 + f_{37} \Delta W_{37}^0 + f_{270}(1 - f_{270})(\Delta W'_{37} - \Delta W_{37}^0)$  where the superscript 0 is used to denote the rate for particles acting individually and the subscript denotes the size. This relation, which requires an accurate measurement of the transient erosion rate  $\Delta W'_{37}$ , is shown by the solid lines in Fig. 7 for

$6 < \Delta W'_{37}/\Delta W_{37} < 8$ , the range estimated experimentally. The results support the predicted trend. It should be mentioned that in an actual service application the situation is most likely to be more complex, due to more complicated particle distributions.

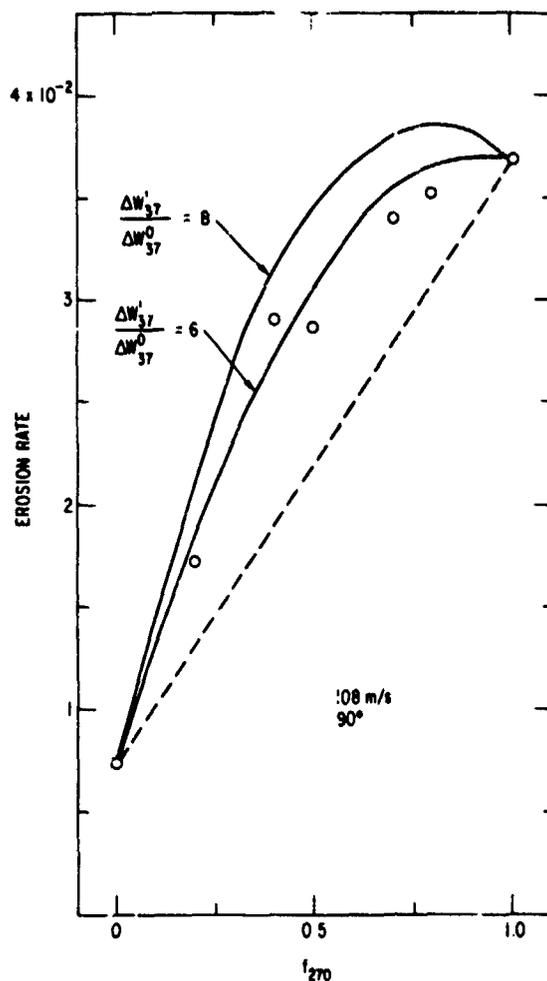


Fig. 7. The steady-state erosion rate obtained for mixtures of 37 and 270- $\mu\text{m}$  particles shown as a function of weight-fraction of 270- $\mu\text{m}$  particles. The dashed and solid lines are explained in the text.

#### V. Summary

The existing models adequately predict the functional dependence, on velocity and size of impacting particles, of the steady-state erosion rates in Si single crystals measured using angular  $\text{Al}_2\text{O}_3$  particles if they are modified to include: 1. plasticity for small impact angles, 2. particle-size (and possibly velocity) threshold effects, 3. a particle-size dependent velocity exponent, and 4. a particle-size distribution effect. The above effects are known to exist, but further systematic experiments are needed to establish the phenomenology in other systems, and

to provide a sound basis for the proper relationships needed in physical models. Theoretical work is needed to incorporate these effects into the models. Synergistic effects are known to exist, but our understanding of them is not complete, and it is certainly not possible to predict complex synergistic effects on the basis of our current knowledge. Finally, the projectile properties (shape and hardness) have never been investigated. Microstructural effects in polycrystalline Si are also possible.

#### Acknowledgements

The authors are grateful to E. W. Kay who performed the erosion rate measurements so capably.

#### References

1. S. M. Wiederhorn and B. R. Lawn, *J. Am. Ceram. Soc.*, 62, 66 (1979).
2. A. G. Evans, M. E. Gulden and M. Rosentblatt, *Proc. Roy. Soc., London*, 361, 343 (1978).
3. T. H. Kosel, R. O. Scattergood and A. P. L. Turner, in Wear of Materials, edited by K. C. Ludema, et al., Am. Soc. of Mech. Engrs., New York (1979).
4. J. L. Routbort, R. O. Scattergood, and E. W. Kay, *J. Am. Ceram. Soc.*, 63, 635 (1980).
5. B. J. Hockey, S. M. Wiederhorn and H. Johnson, in Fracture Mechanics of Ceramics, edited by R. C. Bradt, et al., Plenum, New York (1977).
6. D. B. Marshall, A. G. Evans, M. E. Gulden, J. L. Routbort, and R. O. Scattergood, *Wear* (in press).
7. R. O. Scattergood and J. L. Routbort, *Wear*, 67, 227 (1981).
8. J. L. Routbort, R. O. Scattergood, and A. P. L. Turner, *Wear*, 59, 363 (1980).
9. J. L. Routbort and R. O. Scattergood, *J. Am. Ceram. Soc.*, 63, 593 (1980).
10. D. G. Rickerby and N. H. Macmillan, *J. Mater. Sci.*, to be published (1981).
11. D. G. Rickerby, B. N. Pramila Bai, and N. H. Macmillan, *J. Mater. Sci.*, 14, 1807 (1979).
12. I. Finnie, *Wear*, 19, 81 (1972).
13. R. O. Scattergood and J. L. Routbort, submitted to *J. Am. Ceram. Soc.*

#### DISCUSSION:

BOUJIKIAN: On your formula where  $D_0$  was 12 microns--at that point your erosion sort of stopped--are the particles interfering with each other?

ROUTBORT: No. We are extremely careful to feed very slowly so that we get no interference of particles, in fact single streams of particles. What we cannot do of course, is to get particles 10 microns in diameter to erode. What you can do, however, is to calculate what the theoretical threshold should be and it turns out that in the case of silicon, the threshold is even less than we can measure in velocity. We have to go down to 10 meters per second. There the erosion rate is so slow that we couldn't measure it. So it's not a particle interference effect, it's probably a real threshold effect, but we haven't proved it unambiguously. The material removal rate is proportional to the particle size to the  $2/3$  power, and the velocity anywhere from the 2nd to the 4th power. It depends on particle shape, because that depends on the contact conditions. It depends on the hardness of the material. It depends on whatever model you use, it depends on the acoustic impedance of the target compared to the particle and it depends on the density of the impacting particle.

BOUJIKIAN: The hardness of the particle, therefore, comes into it.

ROUTBORT: The hardness of the target, not the particle. Yes.

CHEN: Your model is based on the complete brittle fracture model, brittle material, no plasticity occurred during the impact.

ROUTBORT: No. That's not quite right. Because you do assume that the kinetic energy of the indenter, if you will, is converted to plastic work in the plastic zone.

CHEN: No, I'm referring to the Weiderhorn paper about six months ago. He used a high-speed camera, and shooting the particle on the glass surface, he definitely showed there's a scooping. Showed the particle really pushed into the glass surface, and melted it...with energy so high it melted the surface and scooped part of the material out.RO

ROUTBORT: Many people observe intense shear zones where there's actually molten material. We have never observed it in silicon. You can indeed calculate that there's enough kinetic energy of the impacting particle to melt the material depending on the conductivity of the material. But we've never seen it.

HEIT: Are the abrasive particles directed against the work in an airstream?

ROUTBORT: No. That's not an airstream. It's under vacuum...the whole system. It's under vacuum because this arm is rotating at 10,000 rpm, and it doesn't rotate very well in air. The particles are mechanically accelerated out the end of the tube.

HEIT: How do you determine the weight loss of the silicon?

ROTBORT: We do it sequentially, we put in a charge of 10 grams, we erode away, we stop, we open the vacuum, we take the samples out and weigh them, we put them back in.

HEIT: Is there any embedment of the abrasive?

ROTBORT: Absolutely none. We've used dispersive X-ray analysis and there's no trace of aluminum; silicon yes, but none of aluminum. Alumina we find embedding in all of our metal work. In fact, many of our metal samples that we've run for various reasons or other gain weight due to embedding. The aluminum and nickel are both fairly soft, the abrasive particle is very sharp, it just sticks in.

WOLFE: I want to congratulate you on a marvelous piece of work that really helps to illuminate what is going on in this silicon removal area. You recommended looking at a higher-density particle like aluminum oxide rather than silicon carbide or such. I think there's probably a small difference. What you did is probably directly applicable to the something like sandblasting, while what we have is a backup of the particles with the tool, so the tool actually imparts the velocity onto the particle and so therefore the density of the particle is probably not as important as its hardness. I think the hardness comes in the size of the impact area. If you have a more ductile particle impacting, the impact area is probably larger, because the particle spreads out. When you have a very hard particle, the impact area is smaller, we have a larger force on a smaller area. I suspect that goes more rapidly to the cutting rate question than the density in this type of cutting we are doing here.

ILES: This is the first paper we've had where people are discussing the mechanics of erosion. It seems to me we've got liquid drops and we've also got particles of silicon from the kerf, coming at very high speed, loose, not bound on the diamond wheel. Are we in the range of speeds where we would expect to see some impact with silicon by silicon itself, which would perhaps modify the cut rates?

ROTBORT: Do you have any idea what the velocities are?

ILES: I suspect it's in the range of 100 meters per second.

ROTBORT: We have significant losses at 10 meters per second with hard particles.

ILES: I'm glad your talk opened up that sort of possibility. That's very interesting.

WOLF: Danyluk's experiments seem to indicate, in light of what you have been showing us here now, that depending upon what kind of lubricant we are using, we could get predominantly ductile erosion, or predominantly brittle erosion. Possibly one kind of hammering of the particles due to some tool vibration and so on, and the other kind, just pushes ductily the material away. Maybe we can learn to take advantage of these.

ROTBORT: These things make a difference of a factor of 4 or so in erosion rate. At least the stuff we've studied. Now four is evidently enough for you people to make big savings.