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High strain rate testing of kidney stones

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Sections of struvite kidney stones were tested in compression at high strain rates ($\sim 3000\,\mathrm{s}^{-1}$) using a Kolsky bar and at low strain rates ($< 0.001\,\mathrm{s}^{-1}$) using an Instron testing machine. The peak stress in both cases appeared to be similar. At high strain rates the values of flow stress measured were between 40 and 65 MPa and at low strain rates they were between 37 and 58 MPa. However, the morphology of the damage was dramatically different. Stones tested at low strain rates formed a small number of cracks but otherwise remained intact at the end of the test. In comparison, stones tested at high strain rates were reduced to a powder. Kidney stones are a two-phase material consisting of a crystalline ceramic phase and an organic binder. We speculate that in the high strain rate tests the large difference in the sound speed between the matrix and the crystalline grains leads to shear stresses that destroy the stone. These data indicate that shear stress induced by the internal structure may be a mechanism by which shock waves comminute kidney stones in lithotripsy.

Introduction

In shock wave lithotripsy (SWL) acoustic shock waves generated outside the body are focused into the kidney and used to disintegrate kidney stones. Since its introduction in the early 1980s, this technique has been the treatment of choice for kidney stones in the United States [1]. Despite the widespread use of lithotripsy the mechanism, or mechanisms, by which the shock waves fragment the stone are still unclear [2]. Further, there is now a recognition that the shock waves induce some trauma to the tissue in the kidney [3, 4]. A better understanding of the interactions of shock waves with kidney stones may provide information that will lead to improvement in the fragmentation effects of the shock waves and reduced side-effects.

In a typical treatment, 1000–4000 shock waves are administered to the patient at a rate of 1–2 Hz. Each lithotripter shock wave consists of a short (1 μs) high amplitude (30–110 MPa) compressive pulse followed by a tensile tail that is approximately 3 μs in duration and has a −10 MPa peak negative pressure. The strain rate induced by the shock wave is on the order of $10^5\,\mathrm{s}^{-1}$. However, the mechanical tests that have been reported on kidney stones to date have been at strain rates of $1\,\mathrm{s}^{-1}$ or less [5]. Thus if one wants to understand the shock wave induced disintegration of these stones, it is important to have information about their response to much higher strain rates.

A number of methods have been developed to test structural materials at high strain rates. One of the most commonly used is the Kolsky or split-Hopkinson bar [6, 7]. Through the use of this bar, which is described in the Experimental section of this paper, one can achieve strain rates between 10^3 and $10^4\,\mathrm{s}^{-1}$. These rates are accomplished by applying the load to the sample through impact. Although these strain rates are not as high as those created during lithotripsy, they should provide much needed information about the mechanical behavior of these stones at higher strain rates.

In this paper, we demonstrate the application of Kolsky bar testing to the determination of high strain rate mechanical properties of kidney stones. Our results show that at high strain rates the stone completely disintegrates into a powder. In contrast, at low strain rates a small number of large cracks formed but the material stays largely intact. This response can be interpreted in terms of the two-phase microstructure of the stone. The data displayed significant variation in the measured flow stress, presumably owing to the natural variability in stone properties and microstructure. However, there did not appear to be a substantial difference in the flow stress between tests performed at high and low strain rates.

Experimental procedure

In order to obtain a stone of a large enough size for the Kolsky bar, we used veterinary kidney stones that are composed of grains of struvite (magnesium ammonium phosphate hexahydrate), which is basically a brittle ceramic, held together by an organic binder [8–10]. Because of their brittle nature, the stones used in this study were found to be susceptible to damage during the

TABLE I Mechanical property data

Stone/sample number	Strain rate (S^{-1})	Maximum strength (MPa)	Notes
1/1	0.001	40	Cross section of test piece 3 mm in diameter
2/1	0.0002	65	1 and 2 taken from the same section of stone
2/2	3000	57	
2/3	3000	58	3 and 4 taken from the same section of stone
2/4	2000	48	
3/1	3500	58	Entire, non-circular cross section of stone tested.
4/1	2500	37	

machining process. The following preparation method resulted in samples with no observable damage. The stones were cut into 4.6 mm thick sections using a high-speed diamond saw. The sections were attached to an aluminum plate using thermoplastic resin and then machined into square pieces. The square pieces were removed from the aluminum mounting plate and bonded to dowel pins using the same thermoplastic resin. They were subsequently ground to cylindrical samples measuring 19 mm in diameter with the original height of 4.6 mm. The samples were soaked in a bath of acetone and placed in an ultrasonic cleaner to release them from the dowel pins and remove any thermoplastic resin attached to the stone. All test samples were prepared in an identical way and were completely dry when tested.

The cylindrical samples were tested in compression at both high and low strain rates. Low strain rate testing was conducted on a screw-driven Instron testing machine. Data were recorded as load vs. crosshead displacement. We did not take into account machine stiffness in converting the displacement data to strain.

High strain rate testing (in the range of 800–3000 s^{-1}) was performed using the Kolsky bar method [6, 7]. This method, in which a material specimen is compressed between two pressure bars, is based on the theory of wave propagation in a bar of elastic material. By measuring the displacements in the pressure bars in response to an elastic stress wave, it is possible to compute the stress and strain within the deforming sample. The two pressure bars, between which the sample is placed, are referred to as the incident bar and transmitter bar. Both have strain gages attached to them, equidistant from the sample on diametrically opposite sides of each bar. A projectile impacts the incident bar, generating an elastic compressive wave within it. A portion of the wave is reflected at the specimen/incident bar interface while the rest is reflected back and forth in the sample to create plastic deformation or is transmitted through the specimen and along the transmitter bar. Because of the difference in diameter between the bars and the sample, the bar can remain elastic while the sample deforms plastically.

The Kolsky bar apparatus used in these experiments had incident and transmission bars that were 3.05 m long and that were made from 6061 aluminum. The projectile length was 0.15 m long and both the projectile and the bars were 0.0254 m in diameter. Strain gages were placed 1.2 m from the sample along both the incident and transmitter bars. The gages recorded the time-dependent strain associated with the reflected and transmitted waves. The strain rate within the specimen was directly proportional to the amplitude of the reflected wave and time integration of this signal yielded the strain. The stress within the specimen was directly proportional to the amplitude of the transmitted wave. Thus, the stress–strain behavior of the specimens was determined solely by making measurements on the elastic pressure bars in the Kolsky bar method.

Results

Table I lists the specimens tested in the experiments along with their testing parameters. In total, four stones were used in this study and in the first column of the table we list the stone number as well as the specimen number for that stone. Specimens 2/1 and 2/2 were neighboring slices of stone 2 and specimens 2/3 and 2/4 were taken from another pair of slices from the same stone. In all other cases we tested only one sample from each stone. No quantitative analysis of the structure of the samples was performed prior to testing. However, for each pair of cylinders taken from a section, care was taken to ensure that the samples had a similar appearance in terms of structure and features. Fig. 1(a) shows an example of one of the specimens. We also examined a stone in the scanning electron microscope, an example of which is shown in Fig. 1(b). The stone appeared to consist of struvite grains that were 20–50 μm in diameter. There was clearly a boundary between these grains, but we could not image this material clearly. However, previous work has determined that these grains are held together by an organic binder [8–10]. We also note that mechanical polishing of the stone prior to taking this micrograph led to surface cracks which can be seen in the figure.

The high strain rate curves for all of the sample showed qualitatively similar behavior. A typical stress–strain curve is shown in Fig. 2. Initially, we see a gradual increase in stress with load up to a maximum of 48 MPa. This increase is more gradual than one would observe for a structural material and we speculate that the slow rise in the kidney stone was due to the formation of cracks. Once the peak stress has been obtained the stress decreases gradually, again in contrast to a structural material which will show a region of plastic deformation. We propose that the decrease in stress in the kidney stone can be associated with the disintegration of the stone into powder. Fig. 3 (right-hand side) shows the stone after testing and illustrates the complete disintegration of the stone during the high strain rate test.

Fig. 4 shows the stress–strain curve for specimen 1/1, which was tested at a strain rate of 0.001 s^{-1}. Note that the curve first increases, hits a small plateau and then increases to the maximum value of 40 MPa. After this

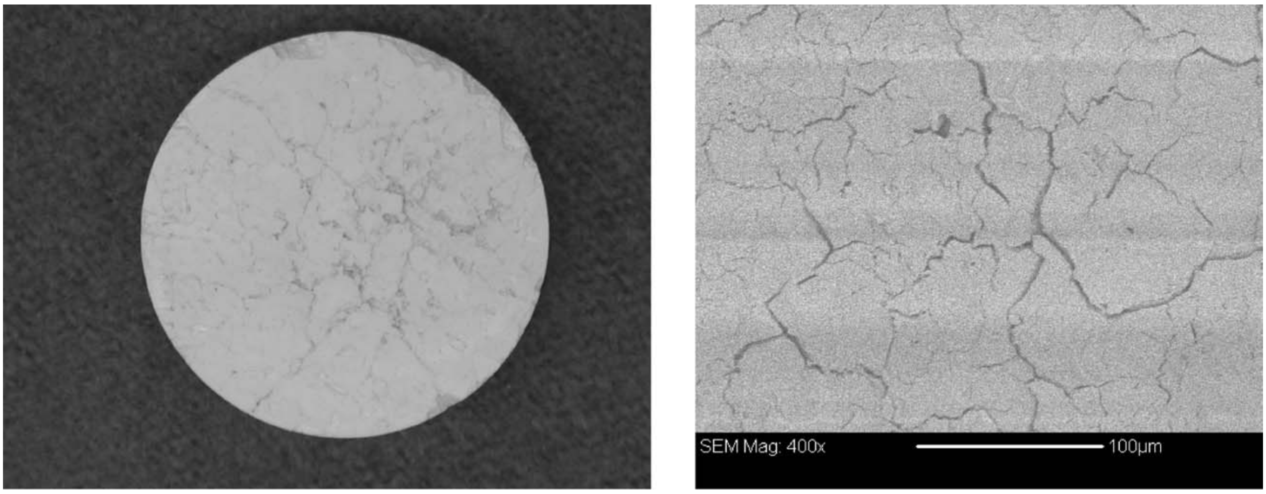

Figure 1 (a) Optical micrograph of a test sample of a struvite stone. The sample is 19 mm in diameter. (b) Scanning electron micrograph of a mechanically polished surface of a struvite stone. The white bar represents 100 µm.

point the stress decreases but never drops below approximately 20 MPa. When the test was complete the sample was still largely intact. An example of a sample tested at a slow strain rate is shown in Fig. 3. Flaking had occurred around the edge of the sample, but it had not disintegrated as had the stone tested at high strain rates. We suggest that in this mechanical test cracks did form within the stone, and these cracks probably began at the first plateau in the stress–strain curve. However, even with cracks present, the stone stayed intact and the long tail at approximately 25 MPa is probably a result of continued deformation of the cracked sample.

Discussion

The results presented above provide information about the response of kidney stones when subjected to compression tests at both low and high strain rates. We now consider these results in the light of other information that is in the literature and discuss why the stones suffered complete disintegration at the higher strain rates.

The kidney stones that we tested had a two-component microstructure. Struvite stones are reported to consist of a ceramic (magnesium ammonium phosphate hexahydrate) in the form of individual small grains, which appeared here to be on the order of 20–50 µm, held together by an organic binder [8–10]. The primary component is the struvite, which is the ceramic and in the form of individual small grains. Upon loading, we would expect the organic binder to yield at a very low stress, but if the struvite is typical of other ceramics, the grains would probably fracture at a high stress with very little yielding. Let us now examine the stress–strain curve shown in Fig. 4 for the test at 0.001 s^{-1}. We see that there is a general increase in the stress with no well-defined yield point. We would expect that as the load is applied the organic binder flows almost immediately and the strength that we measure is primarily a result of the rearrangement and packing of individual struvite grains. The fact that the stone does not shatter comes about from the accommodating deformation of the organic binder that holds the grains together. As mentioned in the results section above, some cracks do form in the stone, and these cracks probably cause the decrease in the stress beyond its peak value. However, the organic binder is able to flow which keeps the bulk of the stone intact and allows it to withstand compressive deformation.

At the higher strain rates, the load is applied through the application of a transmitted stress wave. When the load is first applied, the stress builds up as the stress wave

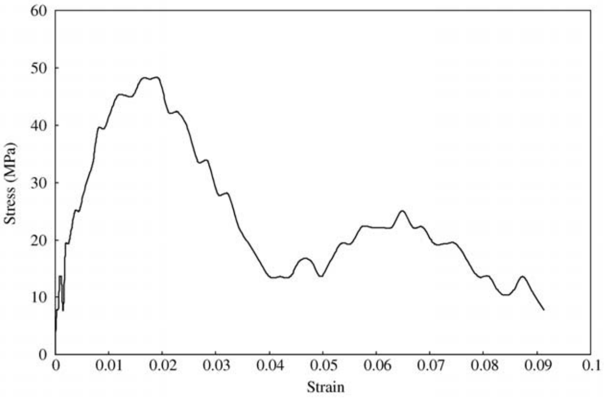

Figure 2 The stress–strain curve for a stone specimen tested at 2000 s^{-1}.

Figure 3 The stone samples after testing. The sample on the left was tested at a strain rate of 0.0002 s^{-1}. Part of the sample flaked off after testing as a result of handling. The powder on the right was produced during a high strain rate test.

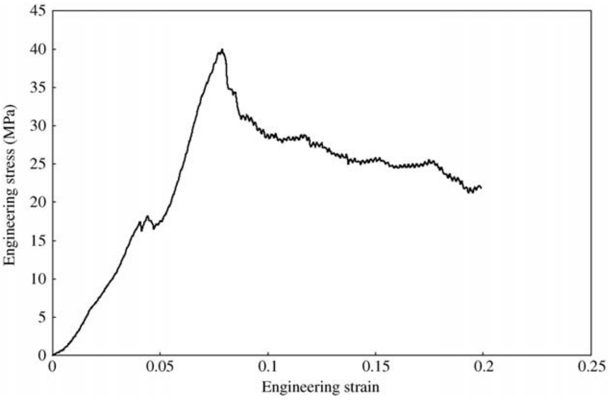

Figure 4 The stress–strain curve for a sample tested at 0.001 s^{-1}.

moves back and forth through the sample. The rate at which this stress increases is directly proportional to the sound speed within the sample. Measurements in the literature indicate that the sound speed is significantly faster in the ceramic part of a kidney stone (3200–3900 m/s) than in the organic binder (860–1450 m/s) [11, 12]. Because of this difference in sound speed there is a significant impedance mismatch at the ceramic-organic interface and shear stress that results from the disparate speeds across the wavefront. Thus, the interface will tend to rip apart, giving rise to the powder that is observed in these tests. The viscoelastic organic materials cannot flow as a viscous fluid at these high strain rates, but rather acts as an elastic solid, which leads to bond rupture within the organic and failure of this layer.

Finally, we note that there have been mechanical tests run on stones of this type at low strain rates. Our peak stresses of 40 and 65 MPa are somewhat higher than the values reported by Ebrahimi and Wang [5], who reported peak compressive stresses on struvite stones that ranged between 10 and 20 MPa. However, the testing done to date in our lab suggests that such variation may result from different microstructures in different stones. Further, Ebrahimi and Wang [5] tested human stones and it is plausible that there is a species dependence of kidney stone properties. Other reports have also emphasized the important effect that different microstructures would have on mechanical properties [13, 14]. For example, Cohen and Whitfield [13] reported observing different microstructural features such as growth centers, inner nuclei and outer shells, and laminations in various types of stones, and they determined that the microhardness varied within these areas. However, we note that in our tests that even though the fracture stress values changed, the difference in fracture behavior remained the same. Thus the observation that the stone stays intact at slow strain rates and disintegrates to a powder at high strain rates results from the fact that it is a two phase material consisting of an organic and a ceramic. The actual values of the fracture stress depend on the details of the arrangement of these two phases.

Conclusions

This note has presented the first results of Kolsky bar tests used to determine the high strain rate response of kidney stones. Our results show that at high strain rates the stone was pulverized into a powder, whereas at low strain rates the stone was left largely intact. The measured flow stress showed a large variation both at low and high strain rate. A statistical comparison was beyond the scope of this study, but data indicate that the flow stress of the Struvite stones tested here was not strongly dependent on strain rate. We interpreted these results in terms of the two-phase microstructure present in these stones. At the high strain rate the organic matrix cannot deform as rapidly as the ceramic; it is torn apart and a powder results. At low strain rates the organic can deform viscoelastically and help bind the material together. Although the fracture stress may vary from stone to stone, the strain rate dependence on the fracture mode appeared to be a general result.

This work was motivated by the use of shock waves to fragment kidney stones. In lithotripsy shock waves will induce a high strain rate in the stones and therefore we expect that the stones will fail in a similar mode to that observed in the Kolsky bar. We propose that as the shock wave propagates through the stone it will propagate more quickly in crystalline grains than in the matrix and the resulting shear stress will lead to fragmentation of the stone. This supposition is consistent with previous data that indicate that the internal structure of kidney stones may play an important role in the destruction of kidney stones [11, 12, 15, 16]. We speculate that this mechanism could be responsible for the large variation in stone fragility seen in human stones as differences in the internal structure would have a significant impact on the resulting shear stresses.

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References

1. K. KERBL, J. REHMAN, J. LANDMAN, D. LEE, C. SUNDARAM and R. V. CLAYMAN, *J. Endourol.* **16** (2002) 281.
2. B. STURTEVANT, in ''Textbook of Endourology'', edited by A. D. Smith, G. H. Badlani, R. V. Clayman, G. H. Jordan, L. R. Kavoussi, J. R. Lingeman, G. M. Preminger and J. W. Segura (Quality Medical Publishing, St. Louis, 1996) p. 529.
3. A. P. EVAN, L. R. WILLIS, J. E. LINGEMAN and J. A. MCATEER, *Nephron* **78** (1998) 1.
4. J. E. LINGEMAN and J. R. NEWMARK, in ''Adverse Bioeffects of Shock Wave Lithotripsy, in Kidney Stones: Medical and Surgical Management'', edited by F. L. Coe, M. J. Favus, C. Y. C. Pak and G. M. Preminger (Lippincott-Raven, Philadelphia, 1996) p. 605.

5. F. EBRAHIMI and F. WANG, *J. Biomed. Mater. Res.* **23** (1989) 507.
6. P. S. FOLLANSBEE, in ‘‘Metals Handbook: Mechanical Testing’’, Vol. 8 (American Society For Metals, Metals Park, Ohio, 1985) p. 198.
7. H. KOLSKY, ‘‘Stress Waves in Solids’’ (Dover Publications, Inc., New York, 1963) p. 30.
8. S. R. KHAN, B. FINLAYSON and R. L. HACKETT, *Scanning Electron Microsc.* **5** (1983) 379.
9. S. R. KHAN and R. L. HACKETT, *ibid.* **6** (1984) 935.
10. K. M. KIM, *ibid.* **4** (1982) 1635.
11. G. PITTOMVILS, J. P. LAFAUT, H. VANDEURSEN, R. BOVING, L. BAERT and M. WEVERS, *Ultrasonics* **33** (1995) 463.
12. G. PITTOMVILS, J. P. LAFAUT, H. VANDEURSEN, R. BOVING, L. BAERT and M. WEVERS, *ibid.* **34** (1996) 571.
13. N. P. COHEN and H. N. WHITFIELD, *World J. Urol.* **11** (1993) 13.
14. A. J. COLEMAN and J. E. SAUNDERS, *Ultrasonics*, **31** (1993) 75.
15. P. ZHONG, C. J. CHUONG, R. D. GOOLSBY and G. M. PREMINGER, *Biomed. Mater. Res.* **25** (1992) 1117.
16. R. O. CLEVELAND, R. A. MULLER and J. A. MCATEER, *J. Accous. Soc. Am.* **110** (2001) 1733.

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