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Dynamic Microindentation of RDX: Effect of Rate on Plasticity and Fracture

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ABSTRACT

Microindentation measurements have been made on RDX crystals to determine the effect of load and time of loading on their mechanical response. A spherical indenter tip was used. The results are compared to those obtained earlier using pyramidal Vickers indenters. At the shortest loading periods a higher than expected plastic response was found which may indicate that local heating resulted from the indentation process.

INTRODUCTION

The uses of energetic materials such as explosives and rocket propellants require materials with controlled ignition behavior. Material characteristics that can lead to pre-ignition are not acceptable. In the case of propellants the material must burn evenly after ignition. There is still disagreement over the basic process of ignition as a result of mechanical impact.

A theory which we support and are investigating together with the University of Maryland (Prof. R. Armstrong) and the Naval Surface Weapons Center (Dr. W. Elban) predicts that impact deformation causes dislocation generation and motion, and subsequent behavior of the dislocations causes ignition [1,2]. Piling-up of dislocations at internal obstacles and their subsequent sudden release are key factors of the theory. Both the kinetic and self-energy of the dislocation substructure are large and either can change suddenly. Concentration of much of the dislocation substructure at barriers or obstacles is postulated to cause formation of local "hot spots" and subsequent ignition.

However, in most materials there are two competing energy dissipation modes, plasticity and cleavage fracture. The role of dislocations in these two modes is different, and the potential for release of energy by dislocations would also be expected to be different. Principal thrusts of this work were to determine the plastic and fracture characteristics, and particularly any rate effects, of a prototype energetic material, RDX. We will report here recent measurements of the response to indentation deformation of RDX (cyclo-trimethylene trinitramine) with respect to the magnitude and duration of loading. Earlier studies using this method have been described [3-5].

EXPERIMENTAL

The NBS Dynamic Microindentation Instrument is uniquely suited to this study. It can work with very small crystals, which are the only available form of production RDX material, and provide mechanical data from within still smaller regions that may correspond to the size of hot spot sites. Further, detailed observations of deformation behavior can be made over a wide range of loading rates even up to impact rates.

The instrument is shown schematically in Figure 1. It uses an electromagnetic driver to force an indenter into the specimen's surface. The loading waveform can be varied in shape, magnitude, and duration. Particular specimen areas can be positioned under the indenter. Since instantaneous values of indenter position and force are recorded during both loading and unloading (Figure 2), it is possible using the resulting load-displacement curve to describe the complete mechanical response of the specimen material [6,7].

Three significant improvements were made recently to the dynamic capabilities of the instrument to better simulate mechanical impact conditions. They required major mechanical and electronic revisions of the instrument. First, dynamic recording capability was extended to shorter times and the lumped load cell/sample resonance was reduced. Samples are now set directly into the load cell. Second, by eliminating the coupled mass of a separate holder, resonance effects are minimized and dynamic range improved. Testing is performed down to 0.001 second duration, which is a 10 fold gain over the previous configuration. The minimum duration limitation is now due to the inertial mass of the indenter assembly. Third, a new depth measuring instrument has been added which has two advantages. Depth sensitivity, volts(out) / µm(displacement), is improved by a factor of 60X; the resolution is now ≤ 0.03 µm. Further the dynamic capabilities are much better; the 3 db band pass limit is 22 kHz rather than 7 kHz. The indenter shape was also changed for this study. A ball, 794 μm diameter, was used rather than a Vickers pyramidal indenter [3]. Advantages of a ball are that it maximizes the tendency of samples to deform plastically, and test results are more easily related to conventional engineering properties. Lastly, by using a spherical shape, the indentation is thought to better correspond to the loading conditions occurring in an RDX aggregate.

Material for the current test series was production RDX (Holston Grade D from Lot #925) which we obtained from the Naval Weapons Center at White Oak. Containing a significant concentration of HMX, production RDX is an impure material. The HMX takes the form of a couplet, a single liquid droplet within a matrix void. No attempt was made to quantify the HMX concentration. Optical examination at 500x magnification revealed a distribution of sizes ranging down to the smallest detectible size. The concentration and void morphology are believed typical of RDX.

When mounting the six (6) different samples into the instrument care was taken to maintain the same crystallographic alignment for all samples. Further, a means of both preparing sample test surfaces and a much improved means of mounting samples into the instrument was developed by recognizing that RDX is hydrophobic. As necessary, test surfaces were lapped flat using water lubricated metallographic polishing wheels. Samples are cemented to the load cell using a water based aliphatic glue. This avoids the contamination which we found organic binders caused, and aids achieving a good coupling of samples to the load cell. Photographs of the mounted crystals are shown in Figure 3.

Three loads and loading durations were used. "Slow" runs had a 100 second duration, "intermediate" runs were 1 second, and "fast" runs had a duration of 1 millisecond. Applied loads of .5, 1, 2 and 3 Newtons were used. Because the 794 µm ball produced broad and shallow impressions not easily observed, indentations at .5 N had no obvious residual impression. Indentation loading times ranged over five orders of magnitude; for each indentation recordings were made of the instantaneous load and depth of penetration.

RESULTS AND DISCUSSION

Representative load-displacement curves for RDX at 1 N load for slow and fast loading durations are shown in Figure 4 and are quite different. The area under the fast curve is greater and the curve is more rounded. The area under a load displacement curve gives the total deformation energy, which can be subdivided into elastic, and plastic components as shown schematically in Figure 5. Table 1 gives individual values measured on the RDX crystals of the three energy components; Table 2 gives the average for each load and time condition. Consistent with Figure 4, we found RDX generally absorbs more deformation energy with increasing rate of loading. The increase is largely due to enhanced plasticity; the plastic energy component increases while the elastic energy component remains nearly constant. These trends are shown graphically in Figures 6-8. The total energy (area under the load-displacement curve) shown in Figure 6 increases slightly with shorter loading times. However, the plastic energy (Figure 7) increases significantly with decreasing loading time while the elastic energy actually decreases slightly (Figure 8). Thus the deformation response to indentation becomes more plastic at shorter loading times. At the highest load of 3 N this trend is different because of the onset of fracture in all the crystals.

A photograph showing a series of seven indentations in an RDX crystal using the spherical indenter is shown in Figure 9. Because of the large radius of curvature, the indentations are not completely visible, rather, the presence of cracks at each indentation is seen in the optical photograph.
In contrast when a pyramidal indenter is used [3], both the indentation and the associated cracking (if any) are readily seen as indicated in Figure 10. It is also possible to detect fracturing associated with indentation from the characteristics of the load vs displacement curve. Figure 11 shows the difference between a plastic response to indentation in RDX and a fracture response; the difference in curves can be understood as resulting from an extended displacement associated with crack growth and perhaps spalling of material in the latter case.

Through microscopic inspection of the indentations and study of the load vs displacement data, we have determined the extent of fracture associated with indentations produced over the range of loads and loading times used in this study. The results are shown in Figure 12 which also includes results from the earlier study [3]. Data were obtained over a range of four decades in load and five decades in loading time. It is seen that three regions exist: (1) where both indenter shapes cause fracture, (2) where only the pyramidal shape causes fracture, and (3) where neither shape does. This is clear evidence for the importance of indenter (or projectile) shape in affecting the mechanical response of RDX and possibly other energetic materials.

Previously we have measured the response of RDX crystals similar to the ones used here to indentation with a Vickers pyramid. Those results, shown in Figure 13, for a slow loading period of 100 s, established the existence of a load threshold for fracture. The curve of load vs diagonal indentation length in the plastic region has a slope of 1.92 which is close to the theoretically expected value of 2.0. In the fracture region, the slope has a much lower value of 0.60. Plain strain fracture mechanics analysis would predict a slope of 1.5. Apparently the fracture process in RDX under conditions of pyramidal indentation is more complex. It also appears to differ from that found [2] in MgO, even though MgO has been used in some cases as a model material for RDX and other energetic solids.

The difference found in response of RDX to mechanical indentation using two different shapes indicates the need for analytical solutions for the subsurface stress and strain fields in this problem. Keer et al. [8] have reported a three-dimensional static solution to fracture under Vickers pyramidal indentation. Because of the observed rate effects and the complex mixing of plastic and fracture response, accurate calculations for a material like RDX would be difficult. An approach similar to that taken by Evans et al. [9] would seem desirable as a first effort. Figure 14 gives a result from that work for the impact of a 400 μm diameter spherical projectile onto a ZnS surface at 860 m/s. It is seen that a large plastic zone is formed of a size comparable to that of the surface crater (200 μm). The impact time interval was of the order of 1 μs. During the unloading period (particle rebound) tensile stresses develop at the edge of the plastic region - in this case as large as 0.5 GPa. Crack growth in these regions would be expected.

The results presented here using a spherical indenter are thought to indicate that near-adiabatic loading of the RDX samples was achieved for the short loading intervals. One would expect that under isothermal indentation conditions, presumably present for the longer loading intervals, any increase in the rate of loading would reduce the extent of plastic deformation and increase the proportion of elastic deformation. However, the opposite was observed in RDX since the proportion of plastic deformation increased with loading rate. In a rate sensitive deformation process the conversion of mechanical energy to heat requires consideration of two factors. One is the effect of loading rate on dislocation mobility, and second is the effect of barriers such as the microscopic regions present in these crystals that contain residual HMX. We believe that the HMX regions can act as obstacles for dislocation glide. Thus in one extreme, at slow loading rates and under isothermal conditions, the dislocations in RDX should be mobile, glide at velocities near the speed of sound, and entrapment times at barriers should be short. Thermal conduction during the relatively long loading period can prevent a temperature rise at the barrier sites. In the other extreme, at high rates, the situation may be far different. Dislocations then spend most of the time trapped at or trying to escape from barriers. In the short time available, conductivity in RDX is too low to quickly dissipate any heating; thus the process is nearly adiabatic in nature. This local heating may be an important factor in "hot spot" formation in energetic materials at high loading rates.

CONCLUSIONS

As a result of this work two important mechanical characteristics of RDX can be recognized. The first involves the importance of indenter geometry. At high loading rates, the plastic behavior of RDX depends on the stress concentration effect of the indenter geometry. Previously [3] we had not found the usual evidence of plasticity (instead finding cleavage fracture), and concluded this was a general property of RDX. This appears only to be true for the Vickers indenter, a high stress concentration geometry. Thus it appears now that RDX is not inherently brittle. Irrespective of the rate with a ball indenter RDX was found to deform plastically for loadings up to 2 N. The second characteristic was that unexpectedly RDX became more ductile under conditions of rapid indentation. This may have resulted from local heating of critical regions in the RDX crystals. Further studies of these rate effects and shape effects would be needed to better understand the material itself.

ACKNOWLEDGEMENTS

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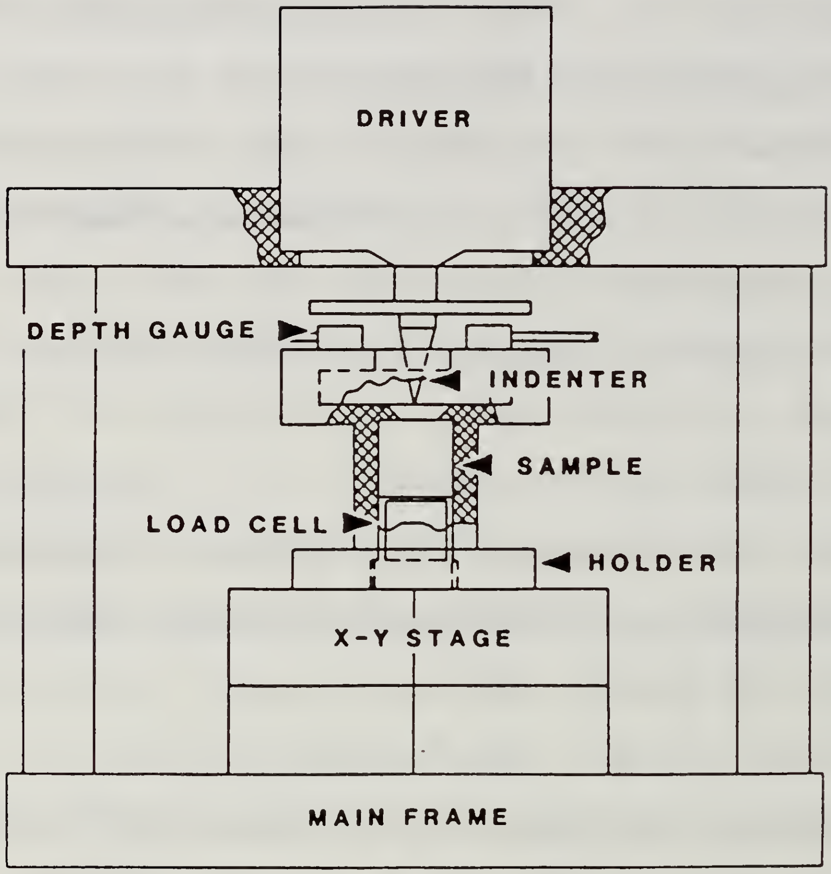


Figure 1. A schematic diagram of the NBS Dynamic Microindentation Instrument.

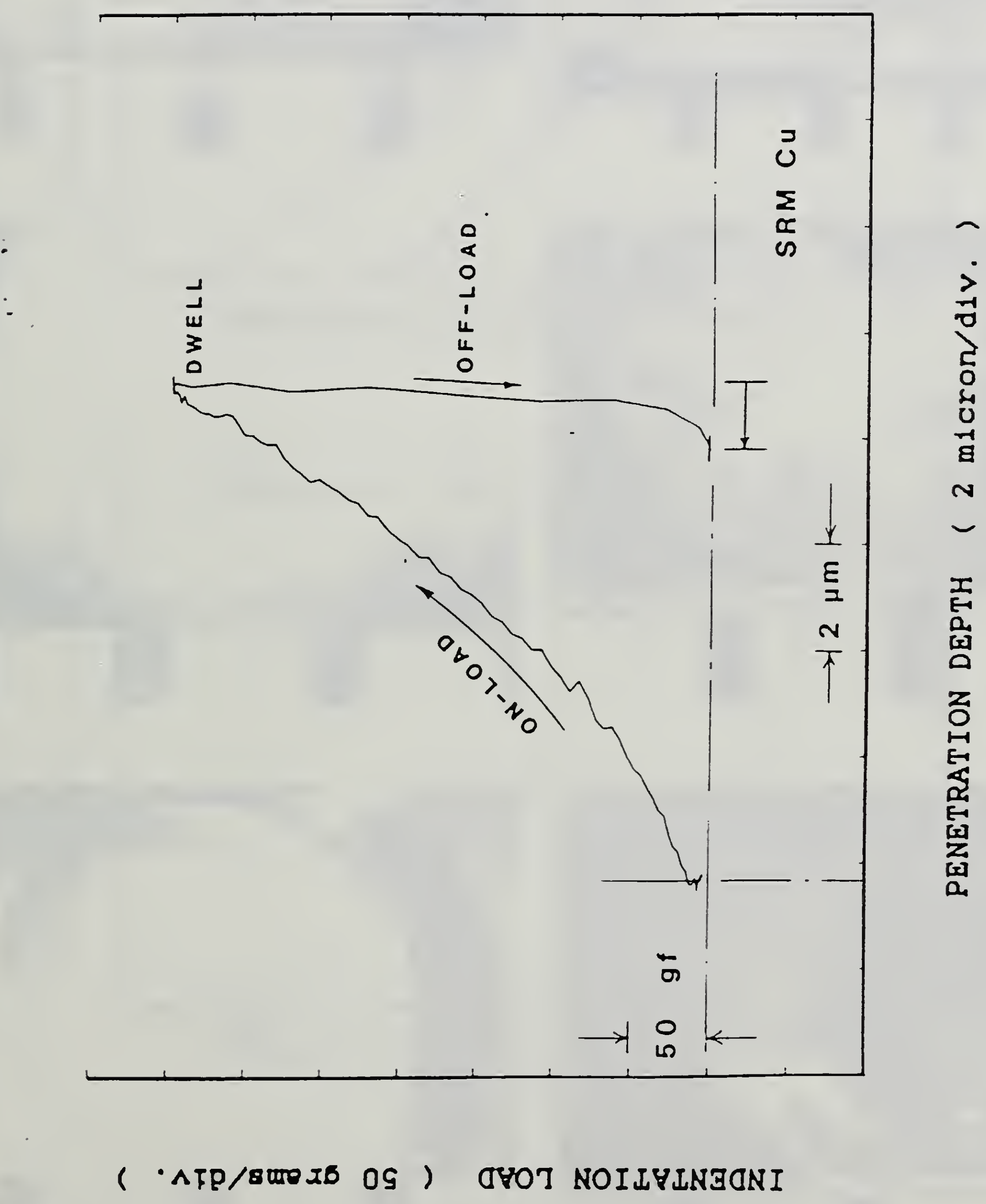


Figure 2. Load vs. displacement curve obtained from an NBS copper microindentation standard material using a Vickers indenter.

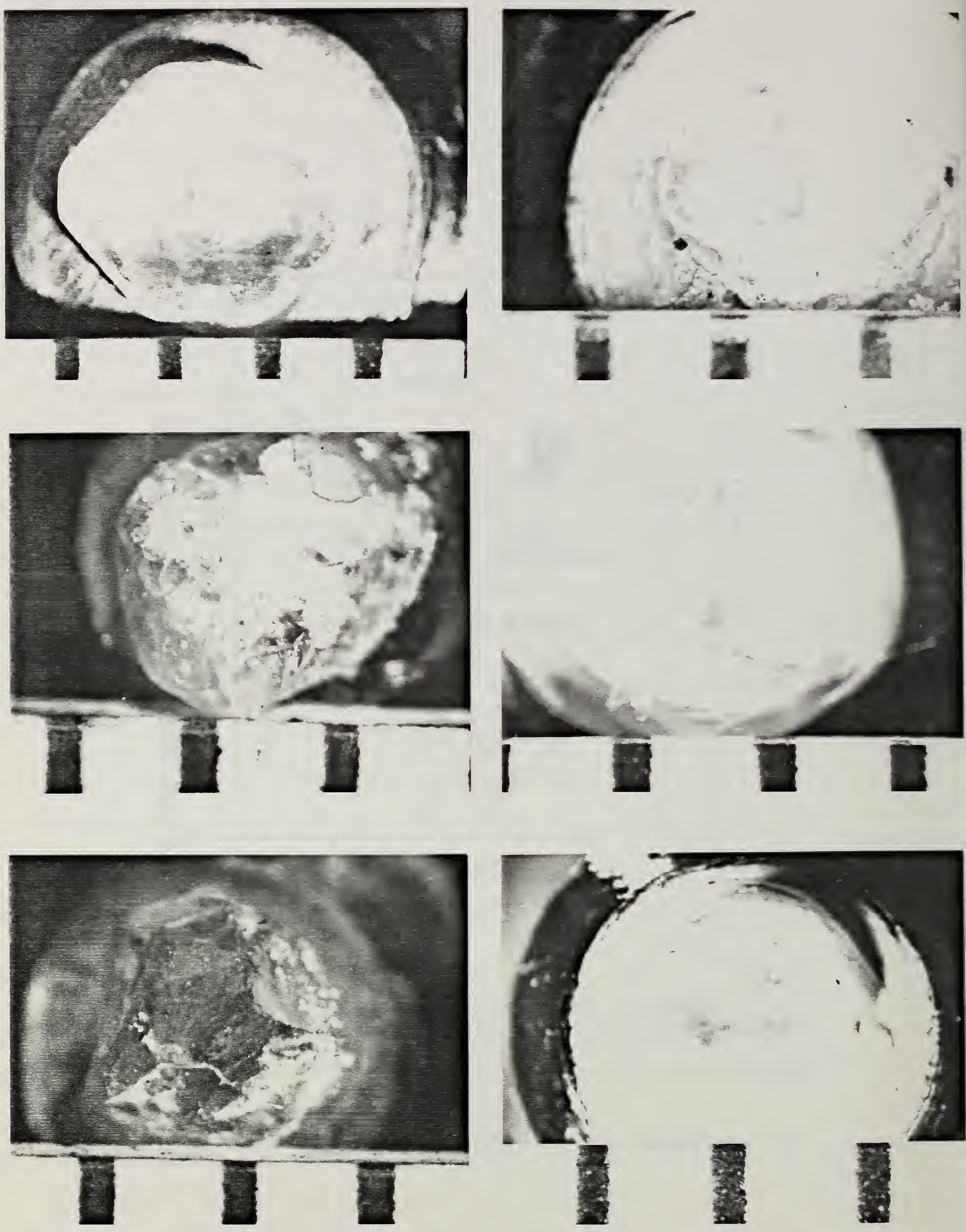


Figure 3. Photographs of the six RDX crystals as mounted and indentation tested. A millimeter scale is also included.

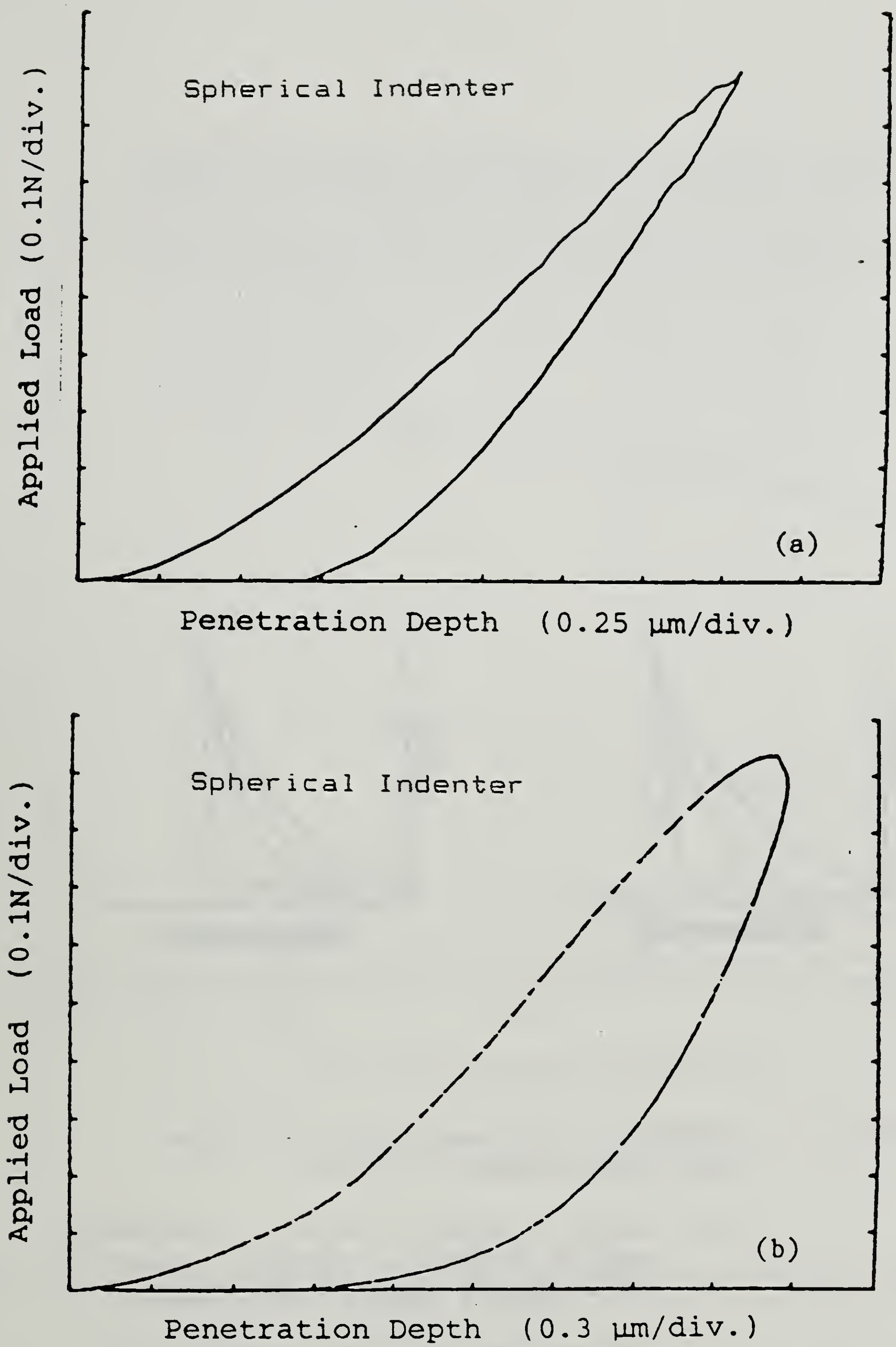
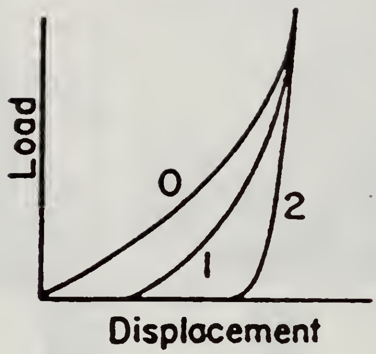


Figure 4. A comparison of representative load-displacement curves for RDX for two loading durations:

(a) 100 seconds
(b) 0.001 second



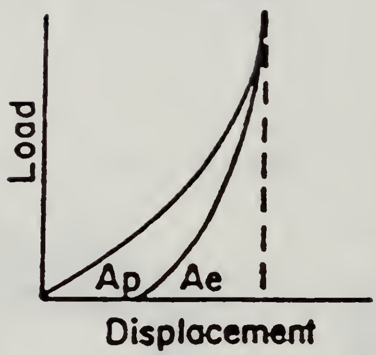


Figure 5. Schematic representation of the load-displacement curve.

(a) After loading, curve 0, recovery can be elastic, curve 1, plastic, curve 2, or some combination.

(b) The mechanical response can be quantified by the relative energy or area under the curves, associated with plastic response, A_p, and elastic response, A_e.

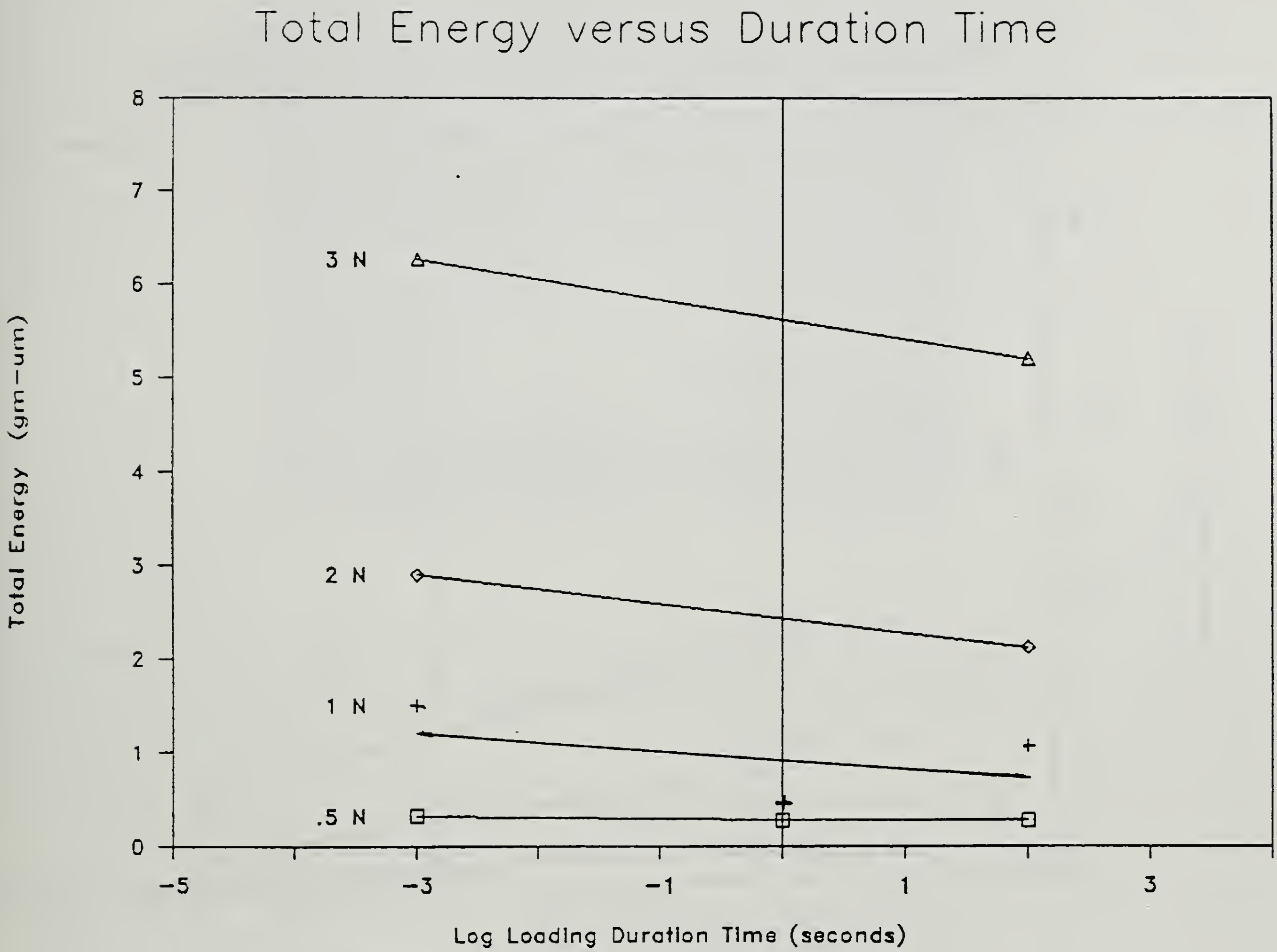


Figure 6. Total energy in g•µm for RDX crystals at indicated loads and loading times.

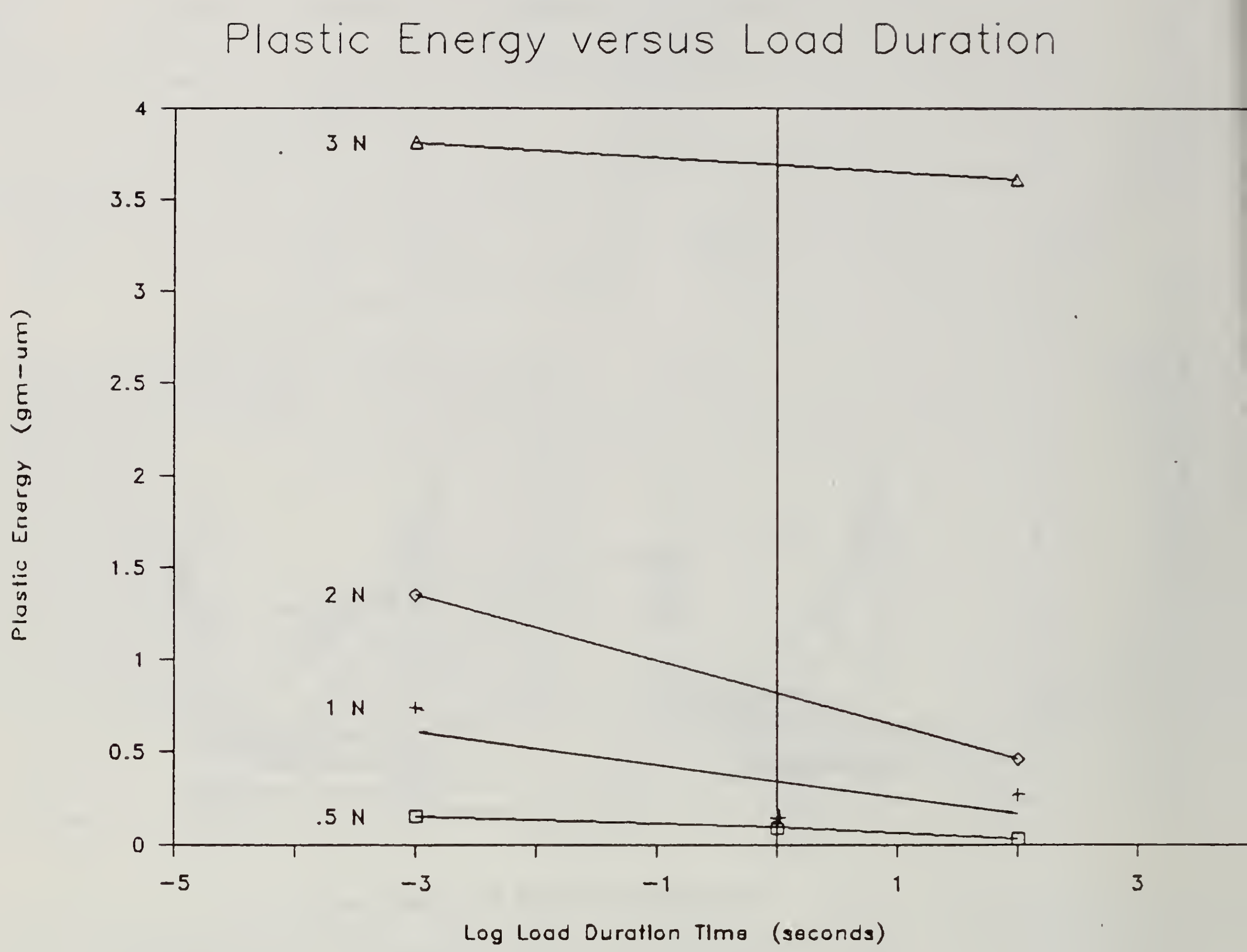


Figure 7. Plastic component of energy in g•µm for RDX crystals at indicated loads and loading times.

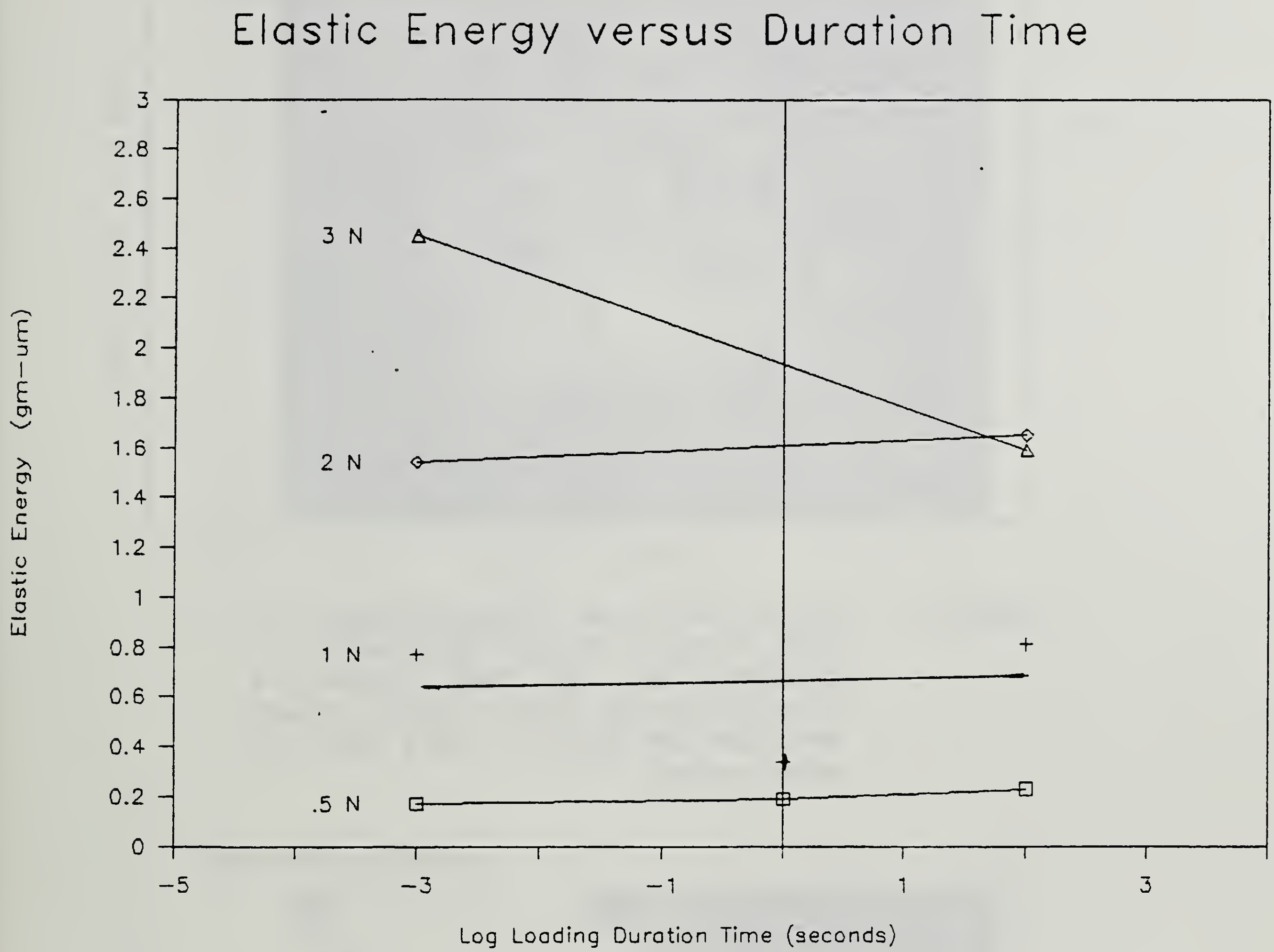


Figure 8. Elastic component of energy in g•µm for RDX crystals at indicated loads and loading times.

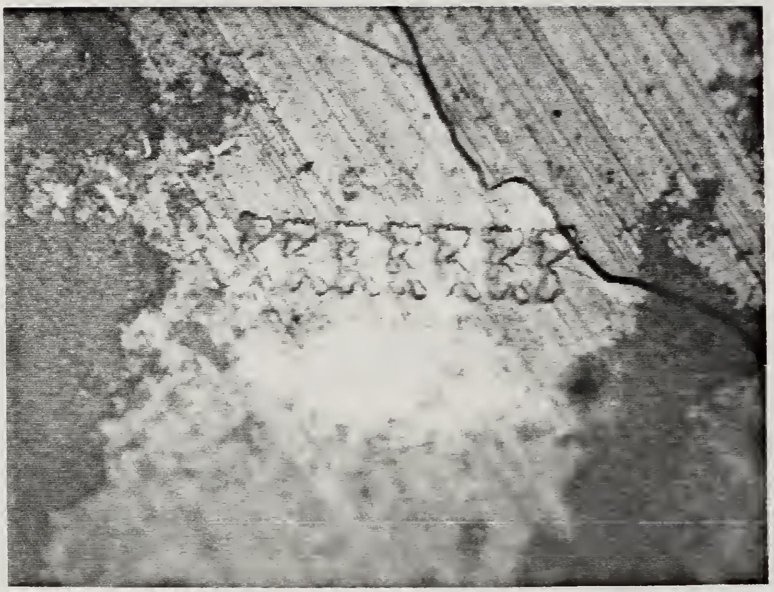


Figure 9. Group of seven indentations on RDX crystal using spherical indentor at 3 N load. Fracture has occurred within each indent and can be seen in this optical micrograph.

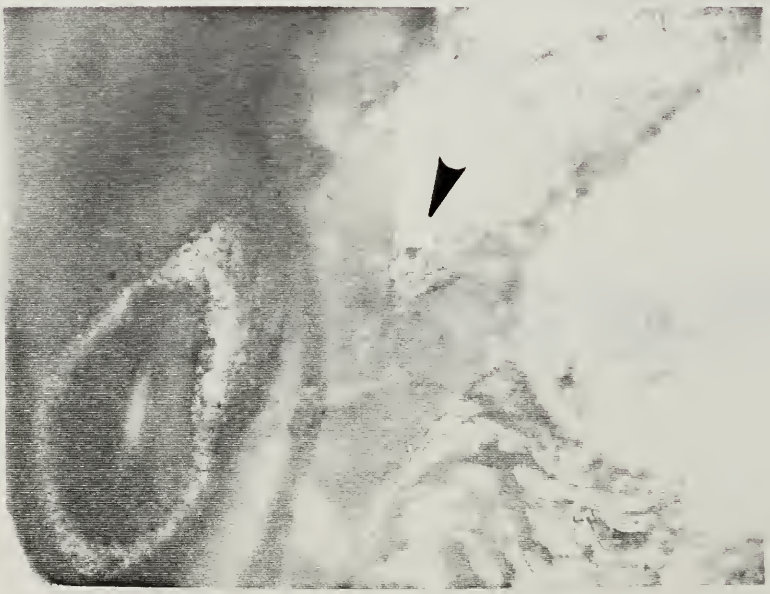
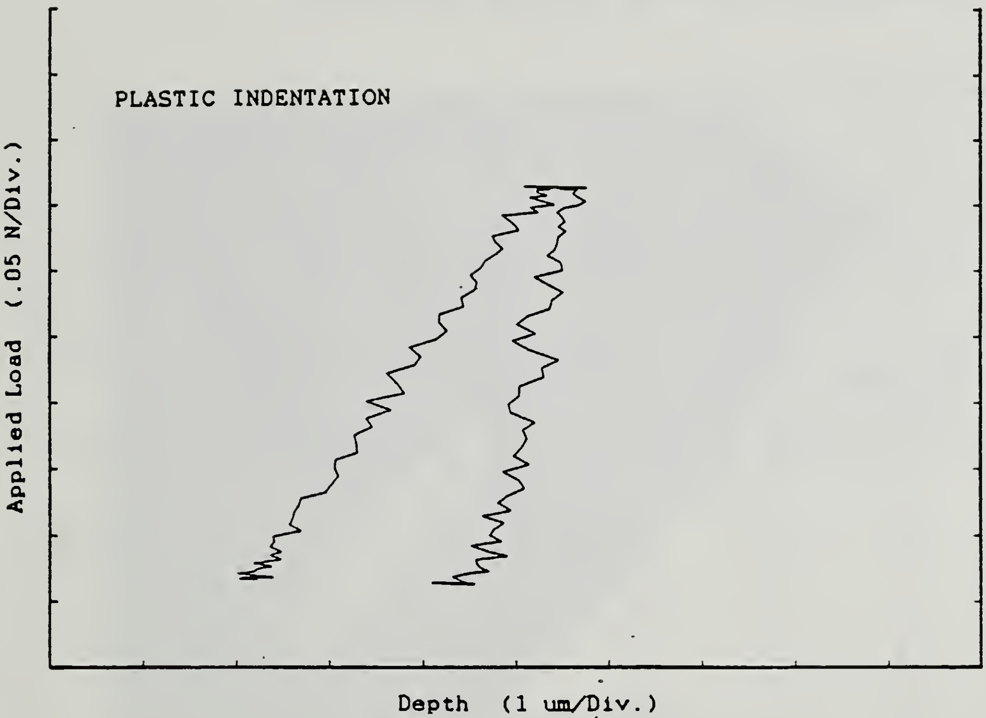


Figure 10. Indentation using a Vickers pyramid (arrow) in an RDX crystal.



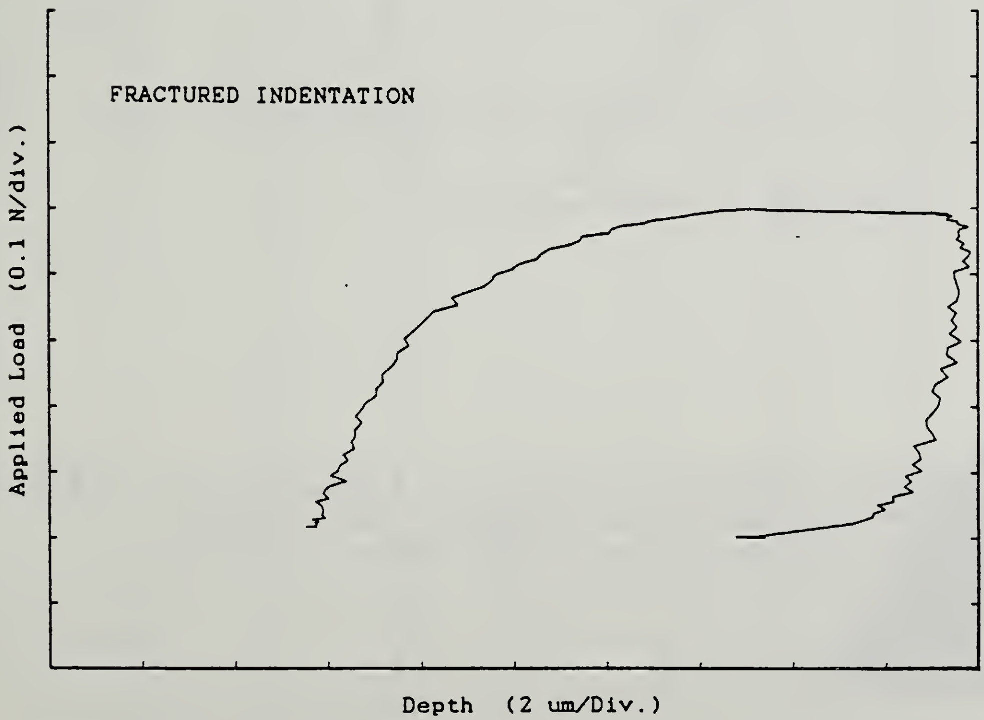


Figure 11. Load displacement curves for an RDX crystal showing the characteristic differences associated with plastic response and fracture response to indentation with the pyramidal indenter.

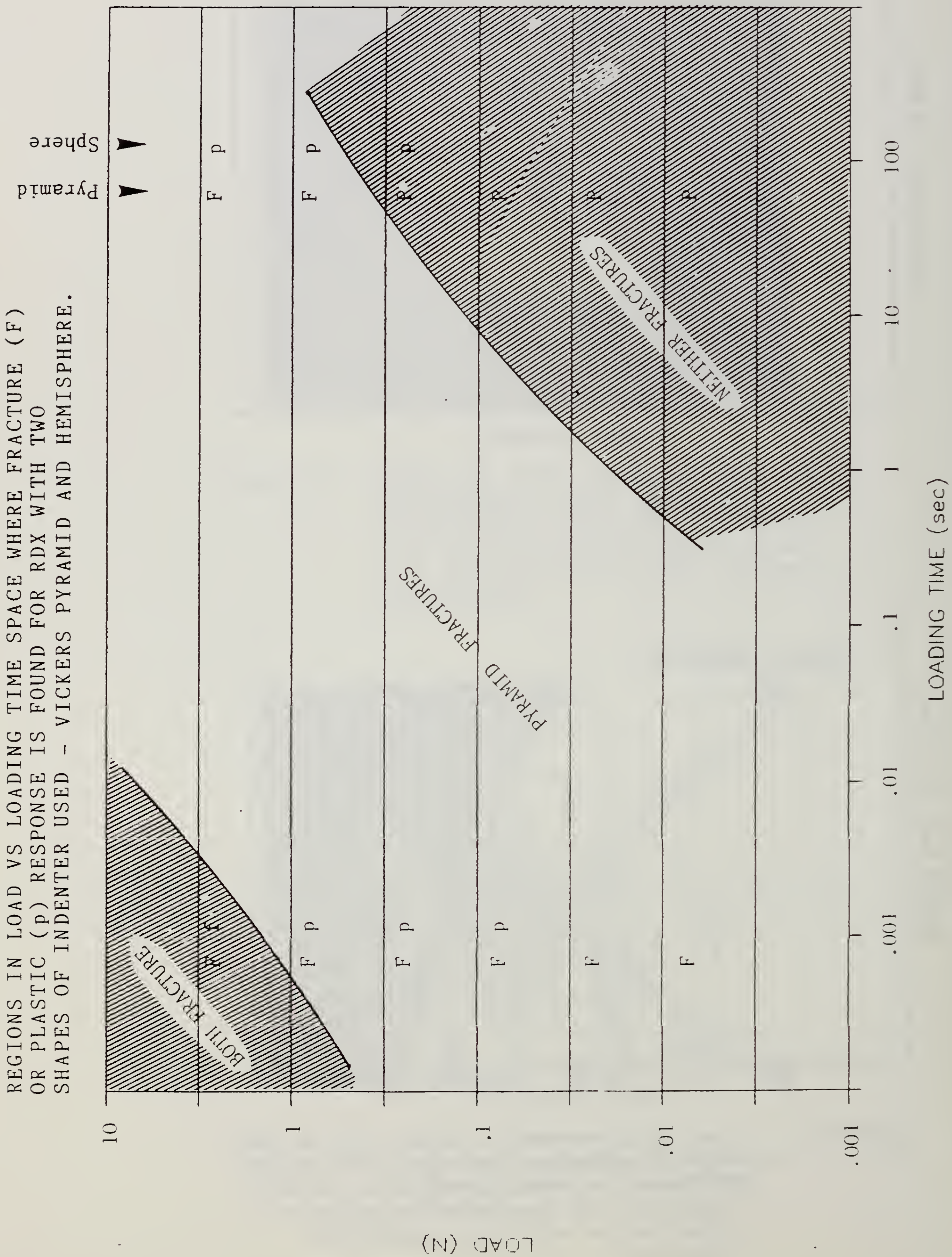


Figure 12. Effect of load and loading time on plastic or fracture response of RDX crystals.

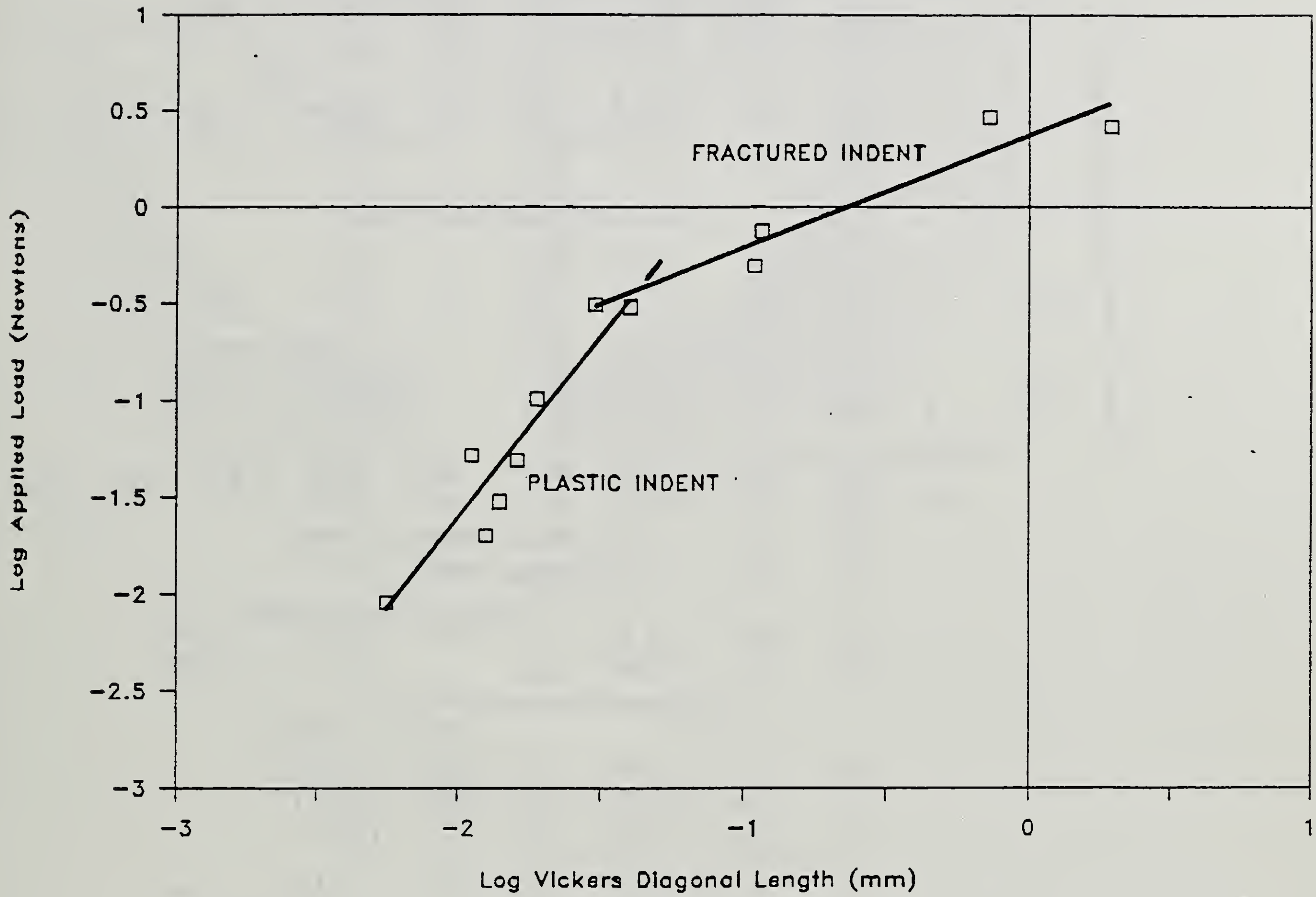


Figure 13. Relationship of load to diagonal length of pyramidal indentation produced in a previous study [3] of RDX crystals under slow loading conditions (100 s).

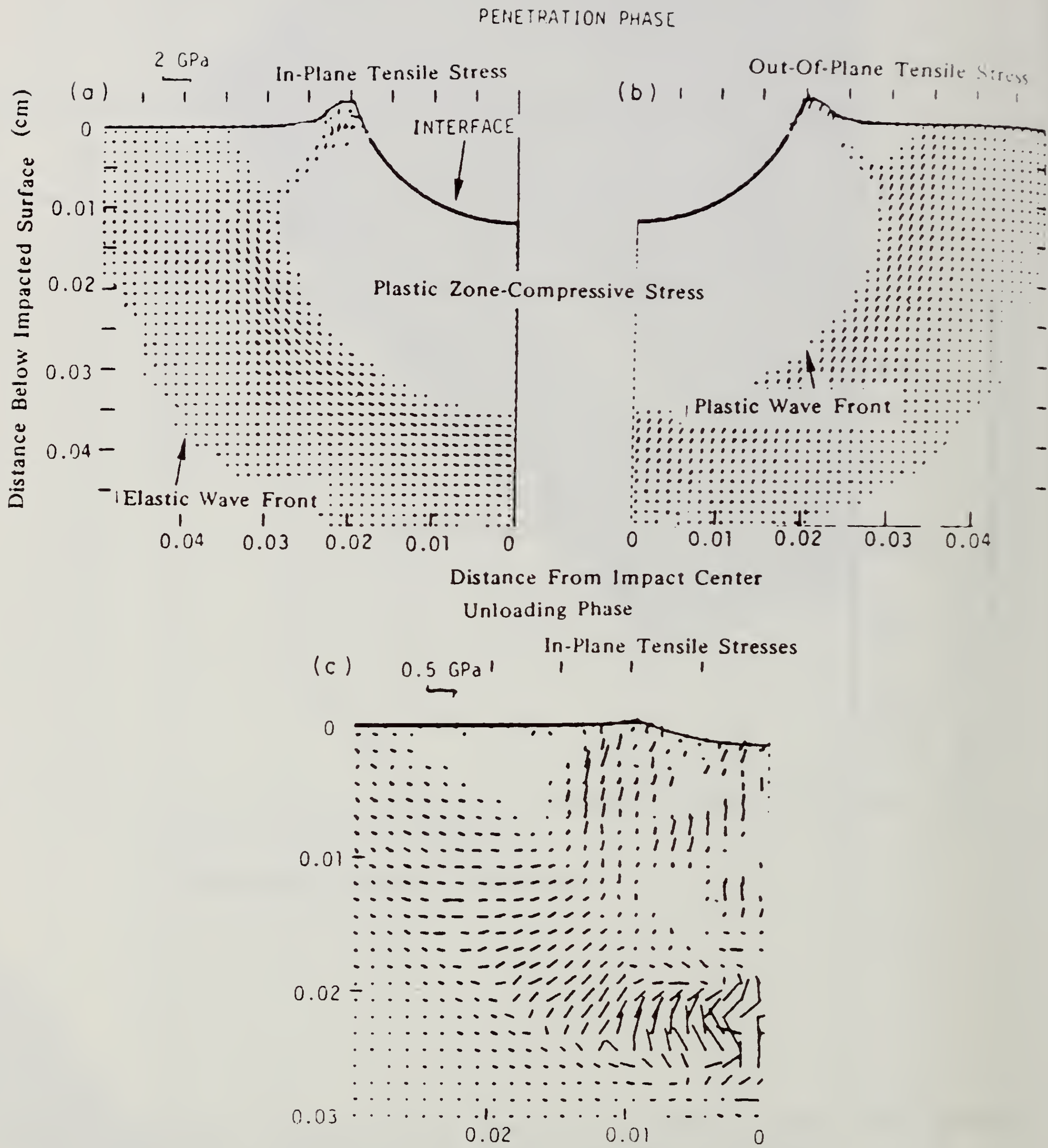


Figure 14. Analytical results of stresses produced by spherical particle impact on ZnS according to Evans et al. [9]. The penetration (loading) phase produces considerable plastic deformation. The unloading phase produces relatively large in-plane tensile stresses. Similar stress patterns might be expected during high rate loading with a spherical indenter.

Table 1. Individual measurements of indentation response of RDX crystals showing elastic and plastic proportions.

	LOAD N	TIME s	ELASTIC um-gm	PLASTIC um-gm	TOTAL um-gm	EL/PL
	0.49	100	0.25	0.03	0.28	8.00
	0.48	100	0.20	0.04	0.24	4.67
	0.48	100	0.26	0.02	0.28	12.73
			------	------	------	------
AVG =	0.48	100	0.23	0.03	0.27	8.46
STD +/- =	0.00		0.03	0.01	0.02	3.31
	0.48	1	0.18	0.10	0.28	1.76
	0.47	1	0.19	0.09	0.27	2.21
	0.47	1	0.19	0.07	0.26	2.79
			------	------	------	------
AVG =	0.47	1	0.19	0.09	0.27	2.25
STD +/- =	0.00		0.00	0.01	0.01	0.42
	0.46	0.001	0.19	0.11	0.30	1.67
	0.40	0.001	0.15	0.15	0.29	1.00
	0.51	0.001	0.18	0.19	0.37	0.94
	0.49	0.001	0.18	0.16	0.33	1.11
			------	------	------	------
AVG =	0.46	0.001	0.17	0.15	0.32	1.18
STD +/- =	0.04		0.02	0.03	0.03	0.29
	1.07	100	0.78	0.29	1.07	2.69
	1.07	100	0.81	0.28	1.09	2.94
	1.07	100	0.83	0.23	1.07	3.61
			------	------	------	------
AVG =	1.07	100	0.81	0.27	1.07	3.08
STD +/- =	0.00		0.02	0.03	0.01	0.39
	0.96	1	0.31	0.09	0.41	3.33
	0.97	1	0.33	0.10	0.43	3.22
	0.97	1	0.38	0.15	0.52	2.56
			------	------	------	------
AVG =	0.97	1	0.34	0.11	0.45	3.04
STD +/- =	0.00		0.03	0.02	0.05	0.34
	1.20	0.001	0.86	0.79	1.65	1.10
	1.14	0.001	0.76	0.63	1.39	1.20
	1.12	0.001	0.75	0.75	1.49	1.00
	1.02	0.001	0.76	0.75	1.51	1.02
	1.17	0.001	0.71	0.76	1.47	0.93
			------	------	------	------
AVG =	1.13	0.001	0.77	0.74	1.50	1.05
STD +/- =	0.06		0.05	0.05	0.09	0.09

	LOAD N	TIME s	ELASTIC u■-g■	PLASTIC u■-g■	TOTAL u■-g■	EL/PL
	1.95	100	1.56	0.46	2.02	3.41
	1.95	100	1.63	0.42	2.05	3.88
	1.95	100	1.77	0.51	2.28	3.47
			------	------	------	------
AVG =	1.95	100	1.65	0.46	2.12	3.59
STD +/- =	0.00		0.09	0.04	0.11	0.21
	2.24	0.001	1.65	1.45	3.10	1.14
	2.01	0.001	1.39	1.06	2.45	1.31
	2.11	0.001	1.58	1.55	3.13	1.02
			------	------	------	------
AVG =	2.12	0.001	1.54	1.35	2.89	1.16
STD +/- =	0.09		0.11	0.21	0.31	0.12
	2.95	100	1.44	3.56	5.00	0.41
	2.95	100	1.74	3.67	5.41	0.47
			------	------	------	------
AVG =	2.95	100	1.59	3.61	5.20	0.44
STD +/- =	0.00		0.15	0.06	0.20	0.03
	2.52	0.001	2.22	3.75	5.97	0.59
	2.44	0.001	2.30	4.00	6.30	0.57
	2.46	0.001	2.69	3.76	6.45	0.71
	2.47	0.001	2.59	3.72	6.31	0.70
			------	------	------	------
AVG =	2.47	0.001	2.45	3.81	6.26	0.64
STD +/- =	0.03		0.19	0.11	0.17	0.06

Table 2. Test parameters and measured energy values for indentation of RDX crystals with hemispherical indenter.

	Load (N)	Time (S)	ENERGY Elastic (µm-g)	Plastic (µm-g)	Total (µm-g)	EL/PL
AVG	0.48	100	0.23	0.03	0.27	8.46
STD DEV			0.03	0.01	0.02	3.31
AVG	0.47	1	0.19	0.09	0.27	2.25
STD DEV			0.00	0.01	0.01	0.42
AVG	0.46	0.001	0.17	0.15	0.32	1.18
STD DEV			0.02	0.03	0.03	0.29
AVG	1.07	100	0.81	0.27	1.07	3.08
STD DEV			0.02	0.03	0.01	0.39
AVG	0.97	1	0.34	0.11	0.45	3.04
STD DEV			0.03	0.02	0.05	0.34
AVG	1.13	0.001	0.77	0.74	1.50	1.05
STD DEV			0.05	0.05	0.09	0.09
AVG	1.95	100	1.65	0.46	2.12	3.59
STD DEV			0.09	0.04	0.11	0.21
AVG	2.12	0.001	1.54	1.35	2.89	1.16
STD DEV			0.11	0.21	0.31	0.12
AVG	2.95	100	1.59	3.61	5.20	0.44
STD DEV			0.15	0.06	0.20	0.03
AVG	2.47	0.001	2.45	3.81	6.26	0.64
STD DEV			0.19	0.11	0.17	0.06

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Dynamic Microindentation of RDX: Effect of Rate on Plasticity and Fracture

5. AUTHOR(S)

R.S. Polvani, A.W. Ruff, and J.C. Robbins

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10. SUPPLEMENTARY NOTES

☐ Document describes a computer program; SF-185, FIPS Software Summary, is attached.

11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)

Microindentation measurements have been made on RDX crystals to determine the effect of load and time of loading on their mechanical response. A spherical indenter tip was used. The results are compared to those obtained earlier using pyramidal Vickers indenters. At the shortest loading periods a higher than expected plastic response was found which may indicate that local heating resulted from the indentation process.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

Dynamic properties, energetic material, fracture,mechanical, microindentation, plasticity, RDX

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