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TECHNICAL REPORT 4311

IMPACT SENSITIVITY OF WETTED PRIMARY EXPLOSIVES AS DETERMINED BY THE BALL DROP TEST

B. D. POLLOCK
R. F. GENTNER

APRIL 1972

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The effects of a number of liquids and liquid mixtures in sensitizing primary explosives were investigated by the ball drop method. Partly wetted lead azides were found to exhibit enhanced sensitivity to impact as compared to like compounds in the dry state. Lead styphnate and tetracene were desensitized by all experimental liquids.

A tentative explanation of this effect is advanced in terms of the role played by liquids in causing desensitization or sensitization, according to the degree of wetting and the existence of confinement which would prevent dissipation of impact energy. According to the model, fully wetted, unconfined explosives are desensitized, but during a drying process, they go through a partially wetted state during which they are more sensitive to impact than when fully dried. Because of the possible value of this model in providing guidance in hazards analysis, it is recommended that the concept be more fully investigated.

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ABSTRACT

The effects of a number of liquids and liquid mixtures in sensitizing primary explosives were investigated by the ball drop method. Partly wetted lead azides were found to exhibit enhanced sensitivity to impact as compared to like compounds in the dry state. Lead styphnate and tetracene were desensitized by all experimental liquids.

A tentative explanation of this effect is advanced in terms of the role played by liquids in causing desensitization or sensitization, according to the degree of wetting and the existence of confinement which would prevent dissipation of impact energy. According to the model, fully wetted, unconfined explosives are desensitized, but during a drying process, they go through a partially wetted state during which they are more sensitive to impact than when fully dried. Because of the possible value of this model in providing guidance in hazards analysis, it is recommended that the concept be more fully investigated.

INTRODUCTION

It is a standard practice to ship primary explosives under a mixture of 50% alcohol-50% water for reasons of safety. It is therefore necessary to dry the explosive before using it, a step that by present methods may require up to 24 hours and gives rise to a considerable inventory of in-process explosive. To increase production rates and simultaneously reduce the in-process inventory, it has been proposed to use a nonflammable, volatile fluorocarbon, such as Freon, as a drying agent to speed up the drying operation. Questions arose concerning the sensitivity of primary explosives to impact while still wetted with Freon.

Studies have been made in the past to evaluate the impact sensitivity of lead azide that has been immersed (rather than being merely wetted or dampened) in various liquids. When Avrami and Jackson (Ref 1) used the Picatinny Arsenal impact machine (Ref 2) and standard impact assemblies to compare the impact sensitivity of lead azide in water, in Freon, and in alcohol-water, they reported an increase in sensitivity as compared with dry azide. They suggested that the liquids play a role in helping to transmit shock to the explosive and raised questions regarding confinement but offered no explanation of the role of that factor. Brown et al (Ref 3) carried out some limited experiments with special purpose lead azide (SPLA) in Freon and also reported an increase in sensitivity in terms of 50% impact energies for azide in Freon that were only one half those for dry azide. Their experiments were carried out using the standard PA impact machine and parts and their azide was also immersed completely in the liquid. They thus confirmed in part the work of Avrami and Jackson.

In later work, Avrami and Palmer (Ref 4) investigated the effects of confinement and wetting by immersion, using Freon, alcohol, water-alcohol, and water as wetting agents. They made use of the Picatinny Arsenal impact machine in combination with special sample containers and strikers designed to permit varying the degree of confinement while the samples were immersed in various liquids. By omitting the liquids, it was possible to make comparisons between dry and wet samples. Sufficient clearance between strikers and sample containers was allowed so the liquid could readily escape from beneath the striker faces. In this sense, the test involved less confinement than exists in the standard PA test. In these tests, Avrami

and Palmer reported that lead azide, when immersed in all the test liquids, showed less sensitivity to impact than when dry, in contrast to the previous work described above.

To resolve the apparently conflicting observations and to provide guidance for the plant modernization program with respect to hazards, it was proposed to conduct a series of tests on wetted, but not immersed explosives using the ball drop technique (Ref 5). This technique has the advantage of being useful for highlighting differences in primary explosives, as well as of economy. In addition, it simulates the hazard situation that is expected to exist. Thus, in both the present and proposed operation, the wet explosive is washed one or more times with liquid and then partially dried by suction on a Buchner funnel (or by some similar suction drying method) so that, for a substantial fraction of the time, the material is dampened but not immersed in liquid. This would likely be the situation also in the event of spills.

The plan of action consisted of testing one lot each of a dextrinated, RD1333, PVA, and Special Purpose type lead azides; one lot of basic lead styphnate; and one lot of tetracene. The liquids of interest in this work were Freon-TF 90%, Freon-10%, ethanol 95%, ethanol 50%-50% water, and water. The mixtures were prepared on a volume basis. A dry sample of each explosive was included as a reference.

EXPERIMENTAL PROCEDURES

The explosives used in this work were all from commercial sources. Lot designations of the azides were: Dextrinated, OMC 69-104; RD 1333, OMC 2-2; and PVA, OMC 69-1. Because there was not enough Special Purpose lead azide in any single lot, a blend was made up from three batches, Dup 53-17, Dup 53-39, and Dup 53-44. The lead styphnate and tetracene lots were OMC 67-2 and OMC 67-18, respectively.

The explosives were stored under a 50% alcohol-50% water solution. For this study, about 80 grams of each explosive were washed, partially dried by suction in a Buchner funnel, and then oven dried for 24 hours at 60° C. The batches were divided into 10-gram portions which were put into conductive rubber containers. These then served as the laboratory supply.

Descriptions of the modified version of the ball drop apparatus and of the test procedures used in this work is given in Reference 5. Briefly, it consists of letting a 1/2-inch-diameter steel ball weighing 8.35 grams fall from a selected height onto a layer of explosive spread uniformly on a hardened steel block. To obtain a thin layer of uniform thickness and of sufficient target area for the ball to impact, the lead anvil block has a shallow groove machined in it, into which about 35 mg of explosive is put with a measuring spoon. The explosive is spread out by running a straightedge or roller on the shoulders of the groove. In this case, the groove was 0.013-inch deep. In the procedure followed in this work for wetted material, the ball was first put into position at the selected height, the explosive was spread out on the block as described and the block put into position below the ball. A few drops of the solvent or solvent mixture was placed by means of a medicine dropper at the edge of the explosive which was immediately wetted as the liquid soaked through. The door of the apparatus was immediately closed and the ball caused to fall.

In the case of the samples wetted with water alone, the procedure described directly above could not be followed because pure water alone did not readily wet the samples. It was necessary to first soak the powder in water, and to then transfer it to the block and spread it in the wet state.

Two groups of tests were run for each explosive-wetting agent combination. The first consisted of a determination of the 50% point by the Bruceton up-and-down method using about twenty-five shots. The second group consisted of sets of twenty shots each, at heights around the estimated 10% point. Four or more such sets were used for each combination. The height for each set was varied in a search pattern analogous to that used for the Bruceton method. This distribution of test heights was selected to emphasize the low probability region, which is of interest for hazards evaluation.

RESULTS

The experimental data for the four lead azides studied in this work is given in Table 2. The points were plotted on probability paper and the best straight lines located visually. These plots are given in Figures 1 through 4. The 10% and 50% heights were taken from these plots and are summarized in Table 1.

The data for both lead styphnate and tetracene in the dry state were treated in the same manner as the data for the lead azides. The 10% and 50% heights for these two cases are as follows:

	10% height, in.	50% height, in.
Lead styphnate	12	15
Tetracene	14	18

Wetting of these explosives appeared to desensitize them markedly. Thus, no fires were observed in ten consecutive trials at the maximum available height, (42 inches), when the explosives were wetted with any of the test liquids. It was therefore assumed that these wetted explosives were desensitized beyond the range of the test apparatus, and accordingly they will not be considered further in this section.

In the case of the dextrinated lead azide (see Table 1), there were no fires in ten trials at 42 inches for samples wetted with alcohol, water-alcohol, and water. This data is treated in the following manner: Assuming the 50% to be 42 inches, or less, then the probability of obtaining ten consecutive no-fires is very low. One is therefore justified in taking the 50% height to be 42 inches or greater. To estimate a probable lower limit for the 10% height, one notes that in the other data in Table 1, the 10% heights are very nearly 2/3 the 50% heights. It is therefore estimated that the 10% heights for these sample-wetting agent combinations are 31 inches or greater. These values are shown in parentheses in the table.

In treating the data in Table 1, it is advantageous to regard the four lead azides as constituting a sampling of lead azides as a class. Thus by averaging the data for these four azides we can deal in a simple manner with the occasional point that appears to be inverted -

the "odd-ball" point - that is so commonly found in sensitivity testing. Further, any conclusions drawn are applicable to lead azides as a group, a useful simplification for guidance in hazards analysis. Accordingly, the averages for the 10% points and 50% points are given in the last double row in the table under the respective liquids. The values in parenthesis next to the averages are the standard deviations among types of azides based on n = 4, or three degrees of freedom for the conditions: dry, Freon, Freon - 10% alcohol. The results for the 95% alcohol and for the 50/50 alcohol/water mixture are combined except that, in both cases, data for the dextrinated azide is not included. This exclusion is considered justified on the grounds that it leads to a more conservative estimate for safety purposes. The averages given under these two liquids are therefore based on n = 6, or five degrees of freedom.

The samples wet with pure water are not considered at this point because of the different sample treatment required to obtain wetting, and are not included in the discussion in the following paragraphs.

The data may now be examined for experimental consistency. First, the 50% height of 23 inches for dry azide obtained in this work is in good agreement with the values for RD 1333 (20 1/4 inches) and for special purpose lead azide (21 - 23 inches) obtained by Smith by the ball drop test (Ref 7).

Next, it may be seen that the ratio of the 50% heights to the 10% heights is very nearly 1 1/2 to 1 in all cases. This finding is in agreement with common experience. Finally, it is seen that the ratio of the heights for the Freon and Freon-alcohol samples to those for the dry samples is, at both firing probabilities, about 1/2 to 1. This finding is in good agreement with the data obtained by Avrami and Jackson (Ref 1) and by Brown, et al (Ref 3) for the case of confined samples immersed in liquid. That is, there is also a corresponding increase in sensitivity for wetted azide in the ball drop test done here.

To test the statistical significance of the differences, the Student t-test (Ref 6) was applied to two pairs, Freon vs dry and Freon-plus alcohol vs dry, for the 10% firing data. The differences of the averages for the first two pairs were found to be large enough to be considered significant at the 95% level. The difference between the water-alcohol and dry samples is not quite sufficient at this level but is sufficient at the 90% level. In view of the fact that the data for the 50%

points shows the same trends in the same relative degree, it seems reasonable to consider that the wetted samples are more sensitive than the dry reference ones.

Even if other methods of testing can show a contrary trend in sensitivity of wetted vs dry or immersed vs dry, one cannot ignore this substantial body of results that clearly indicates that a set of conditions exists where the hazard is substantially greater for wet than for dry azide.

DISCUSSION

This work shows an apparent disagreement with the work of Avrami and Palmer (Ref 4). It may be recalled that they observed a desensitization of lead azide by immersion in Freon, alcohol-water, and water in an unconfined test. In contrast, our results show the opposite effect for wetting by these liquids and mixtures, in an apparently unconfined test.

A point worthy of consideration with respect to explaining Avrami and Palmer's results concerns the behavior of Freon. This solvent contains fluorine and is potentially able to form a thermodynamically very stable fluoride with lead. Thus, because of the exothermicity of the reaction

$$PbN_6 + 2F \text{ (in Freon)} \longrightarrow PbF_2 + 3N_2,$$

sensitization by Freon, to the extent that this factor might contribute to sensitivity, would not have been surprising. However, this factor could not be a consideration for alcohol and water. Hence, the similarity in behavior between the Freon-containing liquids and those not containing Freon in both investigations suggests that the above chemical reaction is not a factor and that it is necessary to look for an explanation in terms of the mechanics of the tests.

It is possible to propose a hypothesis for the apparent discrepancies by examining the role of the liquid in either transferring the impact forces or providing a means for absorbing and dissipating some of the energy of impact. To do this, one must consider the details of the experiments.

First, let us consider the Picatinny Arsenal test used by Avrami and Jackson (Ref 1) and Brown, et al (Ref 3). In the standard test, about 30 mg of granular explosives is put in a small cavity or cup machined in a hardened steel anvil and is leveled off with a spatula, so that the cavity is filled to the top with loosely packed powder. A brass cap fitted over the cup provides confinement. A plug is centered on the brass cap and the drop hammer strikes this plug, which acts much like a firing pin. When the plug is driven down by the drop hammer, it distorts the brass cap and also packs the powder. It has been frequently observed in no-fires that the plug even cuts a disc out of the brass cap. Of the impact is sufficient, the powder fires. It should be noted that, in the standard test with dry powder, there is appreciable "give" in the test fixture, which has a shock-absorbing action.

In the standard procedure used both by Avrami and Jackson (Ref 1) and by Brown et al (Ref 3) to investigate effects of wetting agents, the same amounts of powder were used but before the brass caps were put on, the cavities were filled to the top with liquid. That is, the interstices in the powder were completely filled so that, when the plug delivered its impact to the strongly confined sample, the pressure rise was governed by the high impedence of the liquid and there was no cushioning effect due to powder compression and distortion of metal. One can visualize the particles being subjected to an initial short high pressure pulse followed by other reflected pulses due to impedance mismatch, and also with some focussing due to geometry. Under these circumstances, the explosive could exhibit an increase in sensitivity (as was actually found) due to the pressure-time profile resulting from the conditions of this test.

Next we turn to the later work by Avrami and Palmer on the effect of wetting agents as a function of confinement (Ref 4). For these tests, they devised special sample containers and modified the strikers or anvils used to deliver the impact to the explosives. The containers were prepared from steel cylinders 1 3/4 inches in diameter and 1 1/2 inches high by machining cavities of various diameters and depths. The explosives were put in the bottom of these cavities and, in the wetted experiments, enough liquid was introduced to immerse the explosives to an excess depth of either 1/8 inch or 1 inch. Note that this represents reduced confinement as compared to the previous case.

In one group of experiments of interest to us, the striker was a simple punch with a flat bottom face, which was allowed to rest directly on the explosive. It was held in place by a guide, and the hammer was caused to strike this punch. There was no confinement of the liquid other than that imposed by the excess fluid; that is, high-speed flow of liquid through a porous bed was possible. In all these experiments, a reference set was run by omitting the addition of the liquid, but otherwise maintaining the experimental configurations the same. In all such experiments, the wetted samples displayed less sensitivity than the corresponding dry samples.

An explanation for these results lies in the fact that when the impact is delivered, the impulse is shared between the explosive and the liquid. The liquid in these latter experiments however was free to escape, and therefore provided a shock absorber and lubrication action which alleviated the severity of the impact force as compared to the dry cases. Other experiments were carried out using different shaped anvils and different diameters of cavities, but in each case a means existed for moderating the intensity of the shock when liquid was present, and in each such case the sensitivity of wetted samples was clearly less than that of corresponding dry samples.

Next, consider the ball drop test used in this work. Although this test was originally regarded as being unconfined, a consideration of the details of the wetting procedure and the test geometry suggests that such is not necessarily the case. It is important to note that, in our experiments, wetting was not complete. In all the experiments except those with pure water, capillary action was relied on to cause the liquid to permeate the explosive bed. Only a few drops - less than 0.1 cc - was used. It was not physically possible to completely immerse the explosives, and also the volatility of the liquids made it certain that the interstices in the powder beds were not completely filled with liquid. Because of the absence of a continuous mass of liquid, no shock absorbing action was possible as in the unconfined experiments above.

As to the geometry of the test, it may be recalled that a small ball is allowed to fall onto a bed of loose powder, 0.013 inch deep, and penetrates at least part way into the bed. Although the bed may cover an area of a substantial fraction of a square inch, it can be shown that only about 0.01 inch^2 or less is directly subject to impact;

the rest of the layer provides a sufficiently large target area to ensure that the ball strikes some explosive. Of that material which does participate in the impact directly, only a relatively few grains centered around the vertical diameter of the ball take the brunt of the impact; and the rest acts to provide some degree of confinement. Under such conditions, the available liquid could act to sensitize by providing additional coupling for transmission of the shock of impact between the striker and powder and between powder particles. The sketch shown in Figure 6 may help the reader to visulaize this effect.

A second possible mechanism of sensitization, suggested by Avrami and Jackson, may be due to adiabatic heating of air trapped in the interstices. It is not difficult to imagine that some liquid in the wetted explosive could help trap air more efficiently than would be the case with dry material. It is of course possible that both mechanism could contribute to increased sensitivity.

Some credence for the proposed sensitization mechanism may be derived from results evident in the water-only data, if the greater heights for this set (than for the dry set) are accepted as indicating a desensitization, or at least a reduction of the sensitization shown in the other data. It may be recalled that in order to obtain wetting with water alone it was necessary to completely immerse the explosive in the water and to transfer the explosive to the block in the wet state. Because of the low volatility of water as compared to the other agents, it is reasonable to suppose that in these experiments the explosive was much more nearly completely wetted than in the other cases. If such were the case, the water would provide shock absorbtion in much the same manner as was the case in the experiments of Avrami and Palmer.

To summarize, the results of this work, considered in combination with the findings of previous workers, indicate that sensitization by Freon and alcohol-type liquids is associated both with a state of partial wetting and with immersion. The latter case does not hold for all conditions but, unless one can distinguish the nature of the hazards involved with respect to confinement, one must assume the greater risk, i.e., an increase in sensitivity by immersion.

CONCLUSIONS AND RECOMMENDATIONS

The experimental data obtained in this investigation shows that when lead azide is partially wetted with any of a number of liquids of interest in the plant modernization program it exhibits increased sensitivity to impact. The conditions of the ball drop test are similar to those that might be encountered in practice, where a hard object might drop onto a damp bed of explosive. Prudence therefore requires that damp, but not always fully immersed explosives in a similar configuration. Although the ratio of wetted to dry heights for a given firing probability was found to be about 2 to 1, the importance to be attributed to this difference is a matter of engineering judgment.

Two mechanisms for explaining the effect have been proposed, based on this work in combination with results of previous work. Although the explanations are tentative, they do offer a rational approach and indicate that the mechanism may be applicable to other explosives. It seems wise therefore to consider that the sensitization effect applies to all explosives and to note that, for immersion as well as partial wetting, one must contend with the possibility of increased sensitivity.

If the hypotheses to explain all the observed results are correct, then the present method of transporting explosives (completely immersed in water-alcohol) probably does in fact offer safety so long as care is taken to provide ample excess of liquid and the geometry and nature of the impact are such as to prevent pressure concentrations. One must make sure that, in storage and handling, there is as little confinement as possible. Thus, in the overall drying operation, the sensitivity of primary explosives would appear to proceed from a relatively low value through a state of enhanced (wetted) sensitivity to the sensitivity of the dry state.

The explanation of the sensitization mechanism offered in the Discussion Section of this report is based on limited work, and no doubt numerous challenges can come to mind. However, if the explanation could be verified, it would provide a clear and very useful bit of understanding for use in hazards evaluation. Accordingly, it is recommended that the action of wetted agents be examined more fully in terms of the stress experienced by local sites consisting of powder, liquid, and air in proportions simulating conditions of previous tests and projected plant situations. To this end, it is also

recommended that computer calculations of pressure-time profiles of the standard Picatinny Arsenal impact test be included in such a study. These are essential if impact hazards are to be scientifically forecast.

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7. Joseph Smith, unpublished data

TABLE 1

Impact sensitivity data for wetted lead azides

Lead Azide	Firing level, %	Heights, inches: Dry	Freon	Freon-10% Ethanol	Ethanol 95%	Ethanol-water, 50/50	Water only
Dextrinated	10%	17	8.6	11	≥31	≥31	≥31
	50%	20	19	22	≥42	≥42	≥42
RD 1333	10%	19	7.4	9	11	23	16
	50%	26	10	13	14.6	28	37
PVA	10%	16	5	7	7	10	11.6
	50%	23	9	9	12	21	15
Spec Purp	10%	16	10	11.2	12	10.6	36
	50%	22	12	14.4	16	13	40
Average, and (std dev)	10%	17(1.5)	8(2.3)	10(2.2)	12(5.5)		21
	50%	23(2.5)	12.5(3.9)	15(4.3)	17.5(5.7)		30

TABLE 3

Ball drop impact data
(Height/Percent)

Lead Azide	Dry	Wet With: Freon	Freon/10% EtOH	95% EtOH	50% H_2O/ 50% EtOH	H_2O
Dextrinated	20/50	19/50	24/50	42/50	42/50	42/50
	17/5	9/15	13/10, 13/10	42/10	42/10	42/10
	18/20	10/15	14/15			
		11/15	15/30			
		12/15, 13/25	19/35			
		16/15, 17/30	20/50			
RD-1333	26/50	10/50	13/50	20/50	28/50	38/50
	17/10	6/5	8/10	10/5	22/10	21/25
	19/15	7/5	9/10	11/10	23/10	24/30
	20/10	8/15	10/10	12/35	24/10	26/30
	21/15	9/25	11/20	13/30	25/25	29/25, 30/30
PVA	23/50	11/50	12/50	18/50	21/50	25/50, 15/50
	15/10	5/10	6/5	7/10	10/20	10/5
	16/10, 16/10	6/5	7/5, 7/10	8/15	11/15	11/5
	17/5	7/30	8/20	9/25	12/15	12/15
	18/25	8/45	9/35	10/20	13/25	13/25
		9/50		13/30, 14/55	15/20, 16/25	
Special Purpose	22/50	12/50	17/50	16/50	13/50	40/50
	16/15	9/5	9/10	10/5	10/10	35/5
	17/10	10/5	10/10	11/15	11/10, 11/15	36/15
	18/25	11/15	14/50	13/15	12/25, 12/25	38/20
	19/20	12/40	15/45	14/15		
				15/30		

[a] Indicates percent of 20 samples that fired except that 50% fire point was based on 25 tries. Heights are in inches.

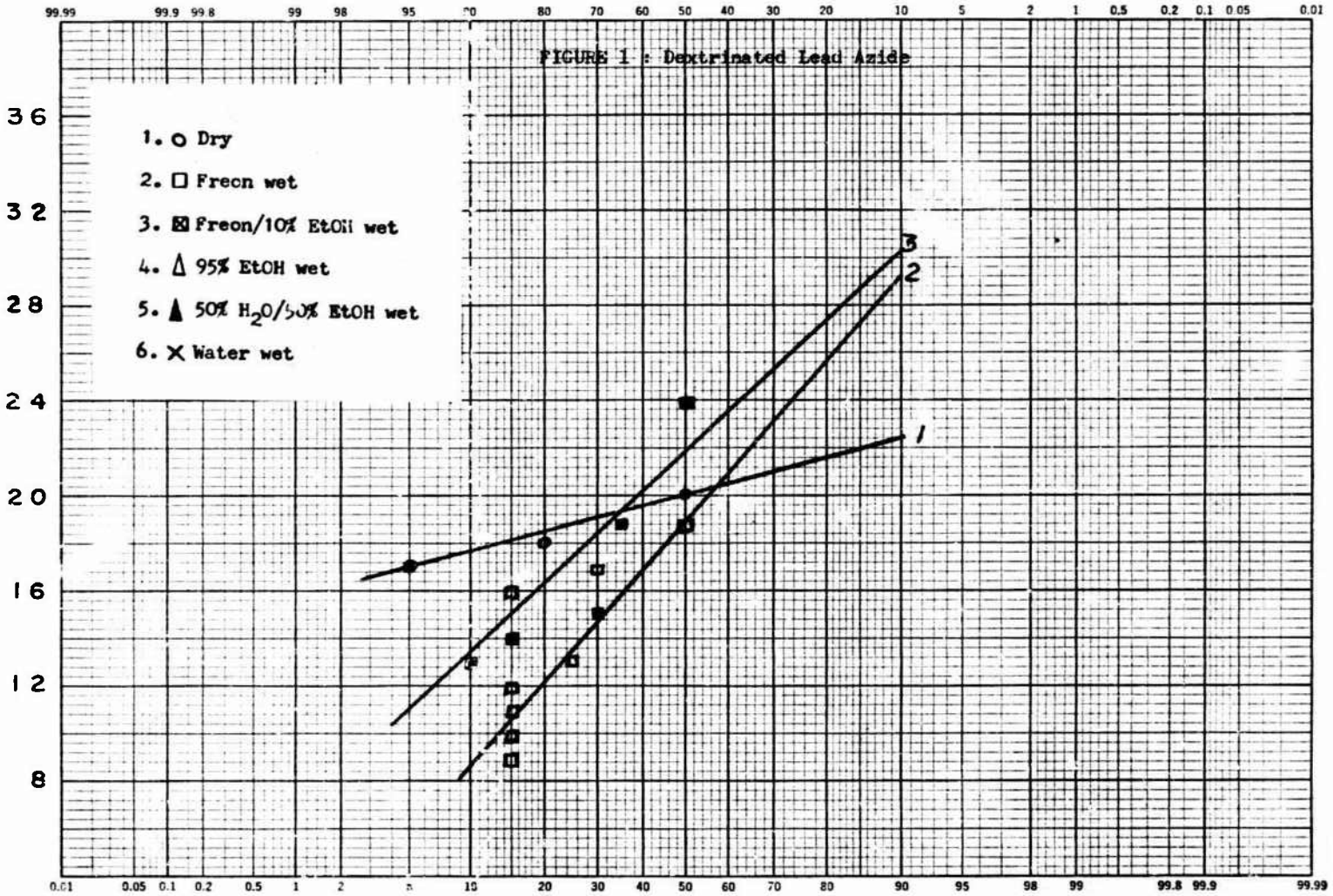

Fig 1 Dextrinated lead azide

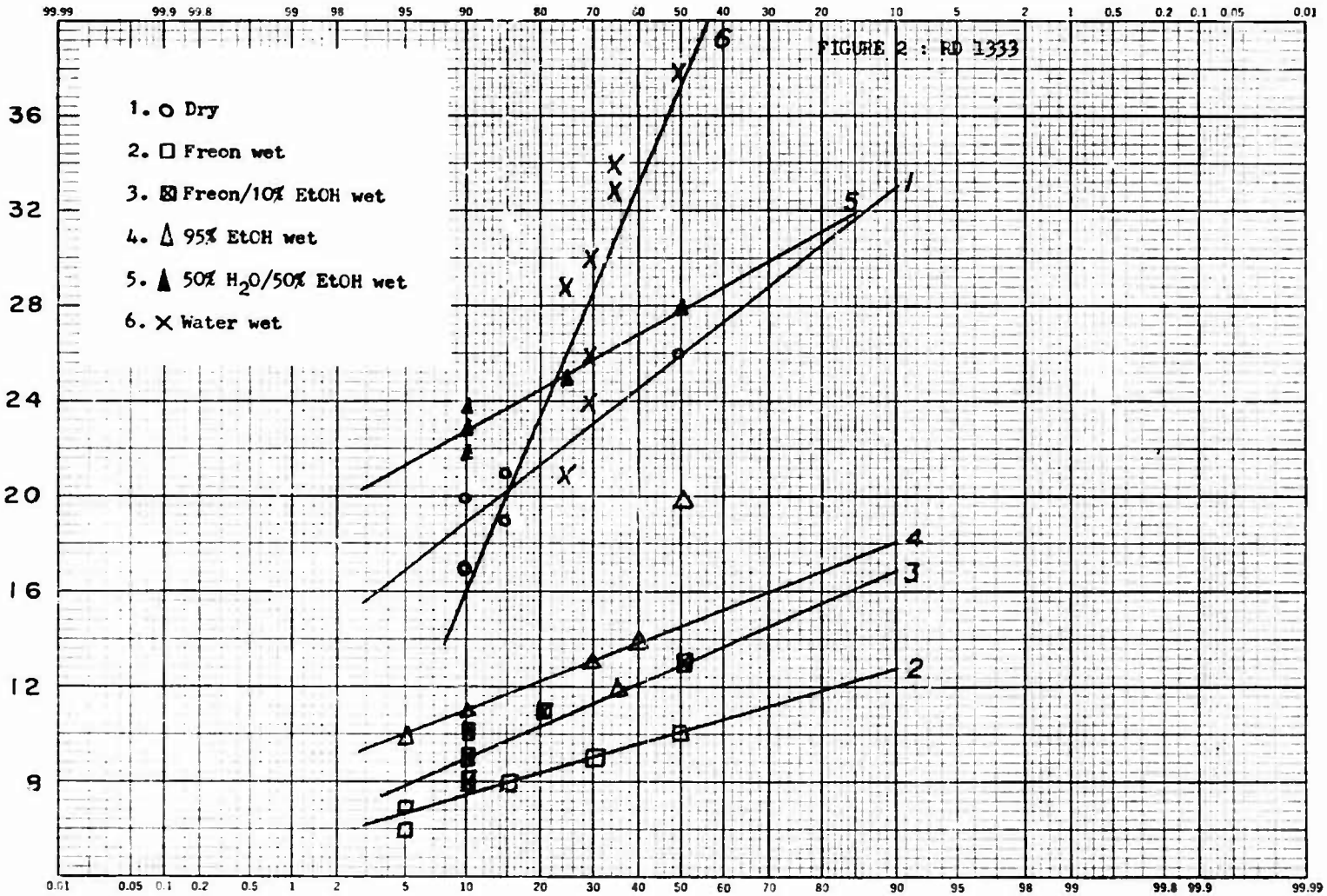
FIGURE 2 : RD 1333
1. Dry
2. Freon wet
3. Freon/10% EtOH wet
4. 95% EtOH wet
5. 50% H_2O/50% EtOH wet
6. Water wet
36
32
28
24
20
16
12
9
99.99
99.9
99.8
99
98
95
90
80
70
60
50
40
30
20
10
5
2
1
0.5
0.2
0.1
0.05
0.01

Fig 2 RD1333

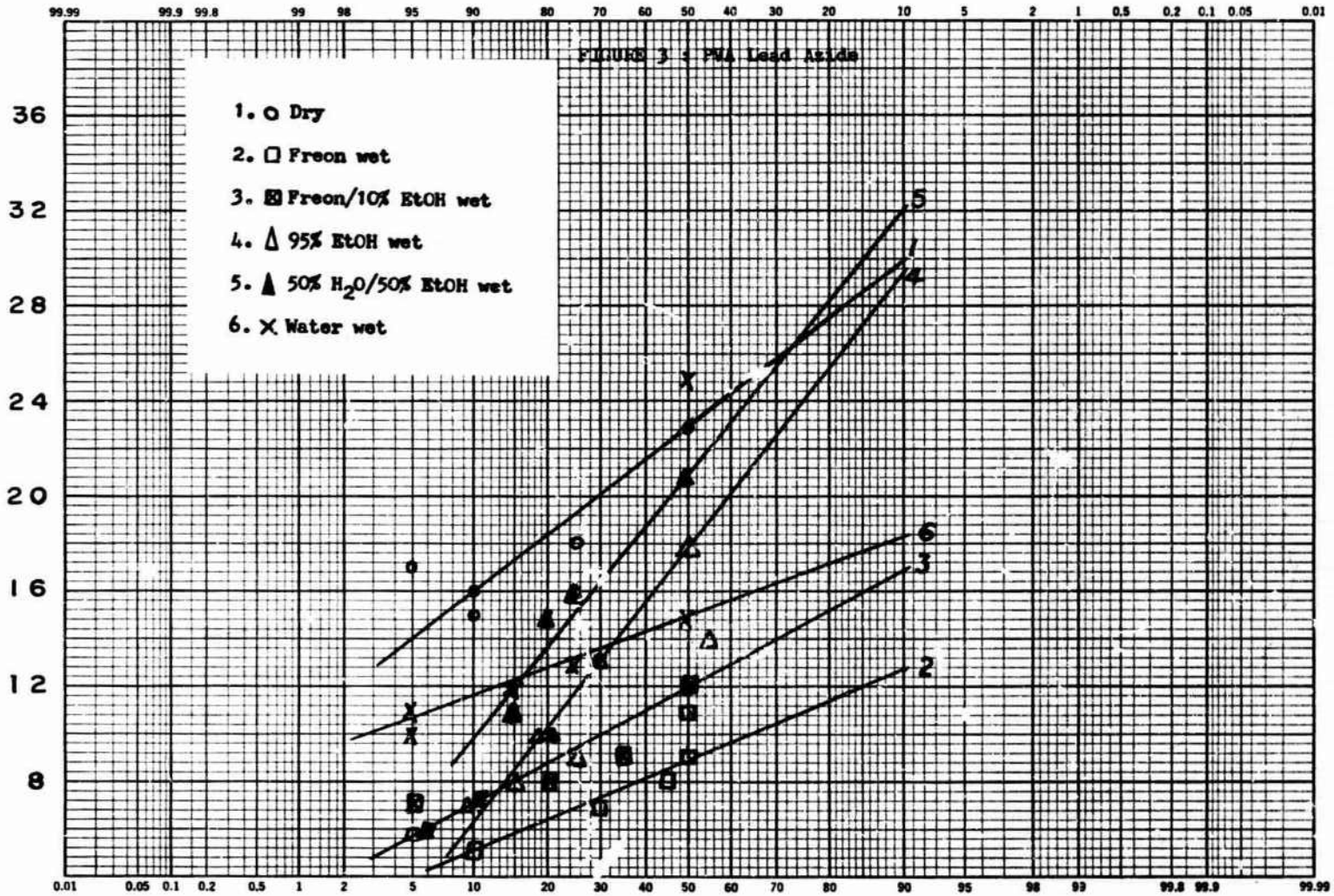

Fig 3 PVA lead azide

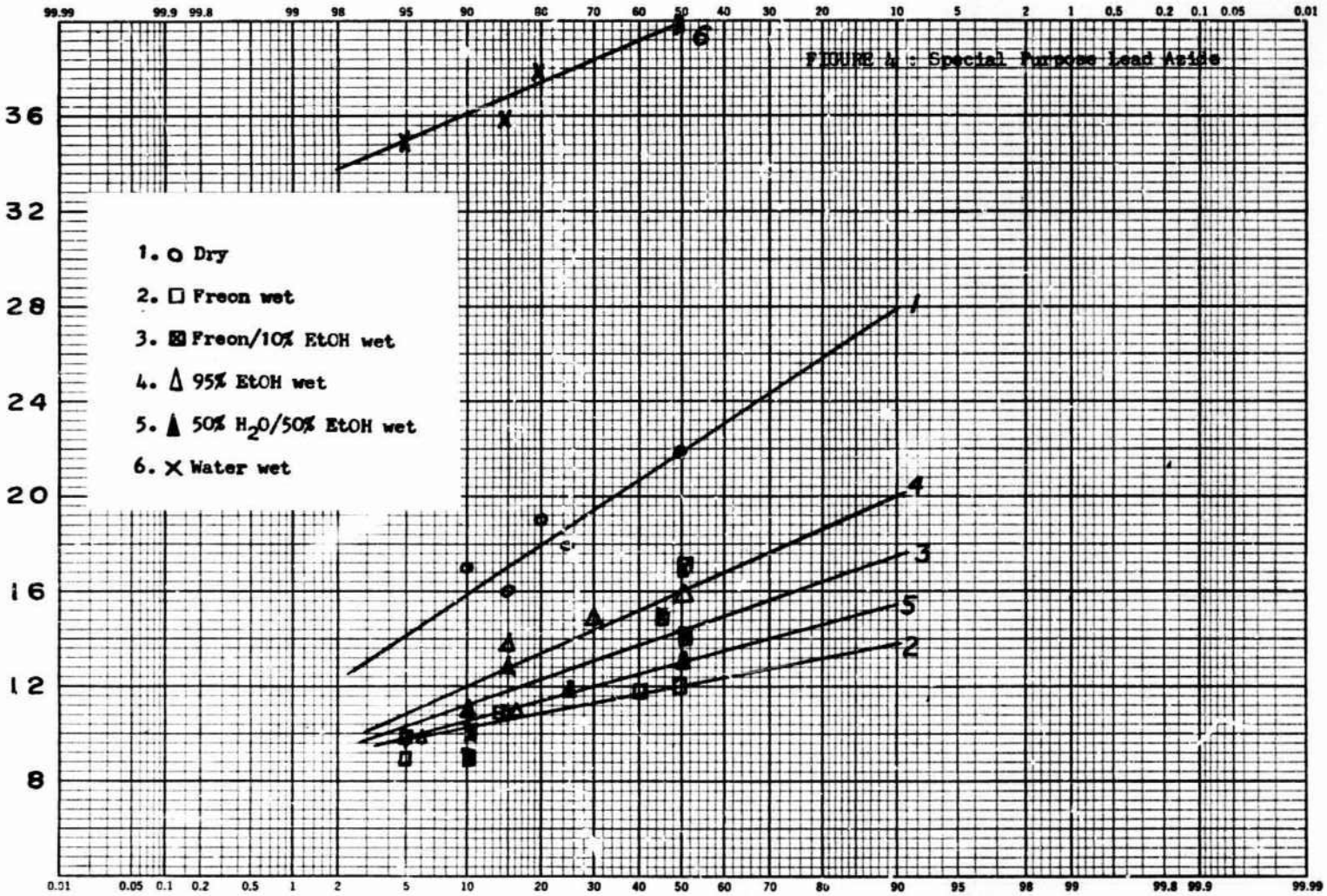

Fig 4 Special purpose lead azide

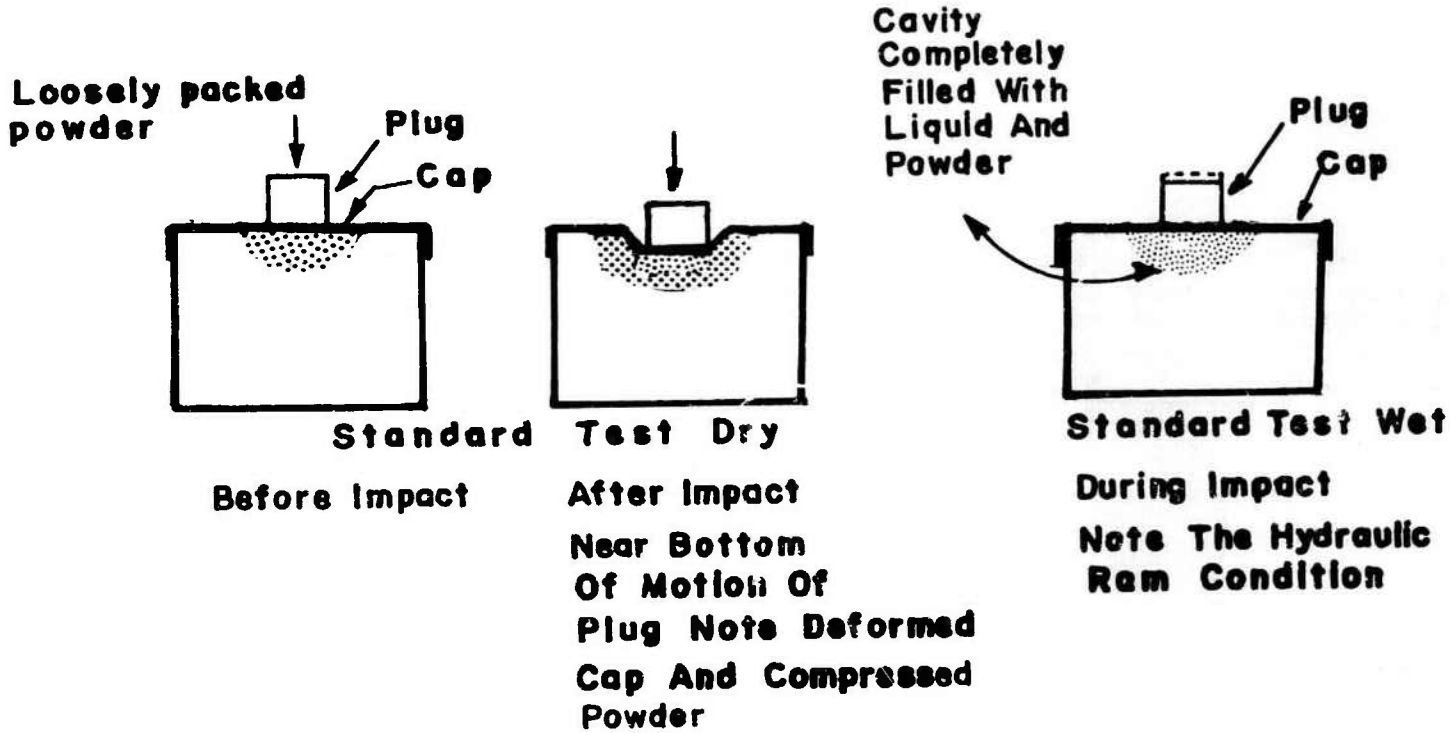

Fig 5 Dynamics of the Picatinny Arsenal impact test

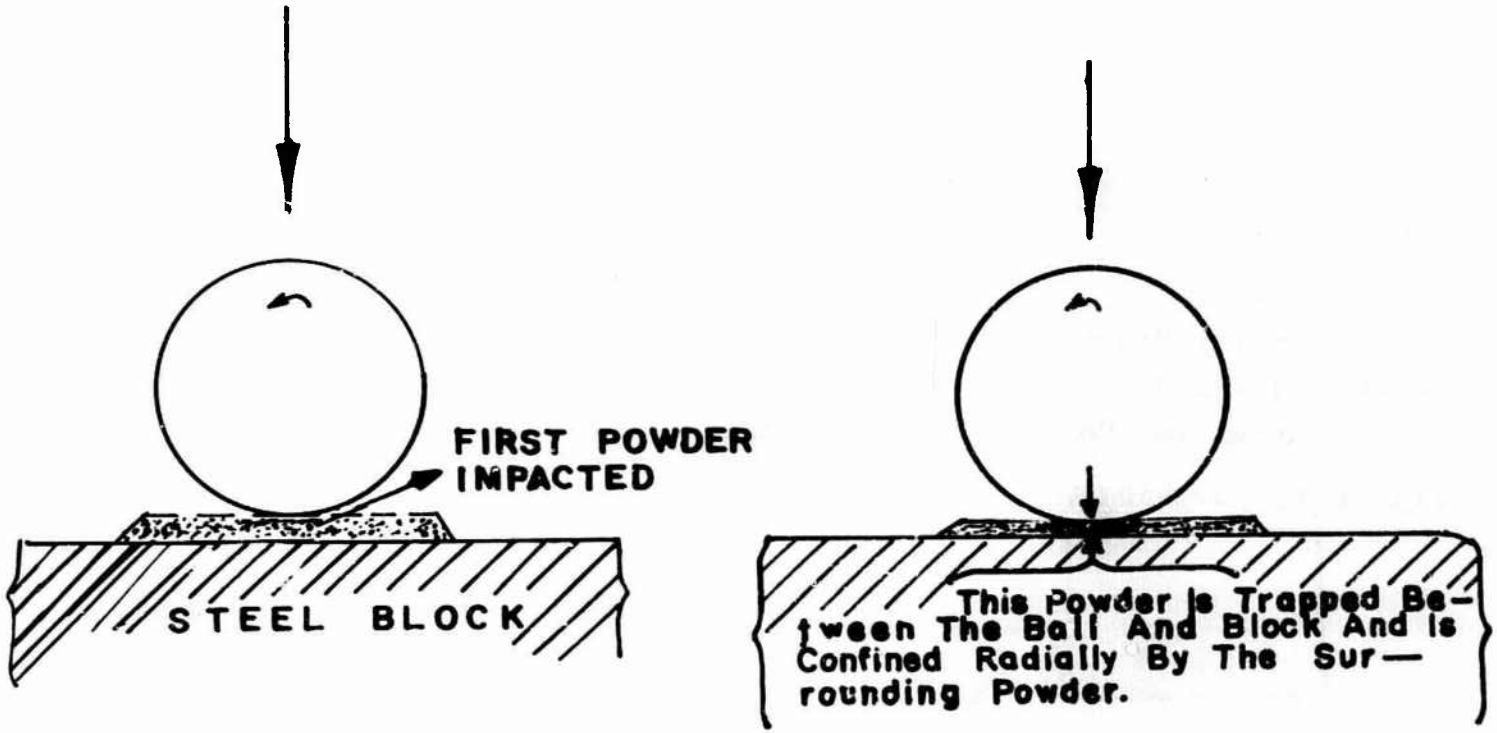

Fig 6 Dynamics of the ball drop test