FOREWORD

This report was prepared by Nitrogen Division, Allied Chemical Corporation under USAF Contract No. AF 33(616)-7114. The contract was initiated under Project No. 7312, "Finishes and Materials Preservation," Task No. 73122, "Corrosion and Deterioration Control." The work was administered under the direction of the Materials Central, Deputy for Advanced Systems Technology, Aeronautical Systems Division with Mr. Harold L. Stevens acting as project engineer.

This report covers work conducted from April 1960 to April 1961.

The work was done in the Corrosion Laboratory of Nitrogen Division, Allied Chemical Corporation, Hopewell, Virginia. In addition to the authors, Mr. John L. Grinstead assisted in the experimental work and Mr. James D. Ashton supervised the construction of the apparatus. Acknowledgement is made to Mr. R. C. Datin for advice and guidance in carrying out the work.

ABSTRACT

The purpose of this investigation was to determine the impact sensitivity of commercially pure titanium, titanium alloy 6Al-4V, and precipitation hardened 15-7 Mo stainless steel when exposed to liquid nitrogen tetroxide. An explanation is given of the probable mechanism of the limited ignition resulting from impact.

Commercially pure titanium and titanium alloy 6Al-4V ignite about 50% of the time when impacted with a 0.5 inch diameter flat striker having an energy of 200 ft-lbs. Storage of the commercially pure titanium in N_2O_4 decreases the energy needed for 50% ignition to 60-70 ft-lbs. The sensitivity of the alloy is unaffected by storage in N_2O_4.

Precipitation hardened 15-7 Mo stainless steel was not impact sensitive in liquid N_2O_4.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

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1. INTRODUCTION

1.01 In the rocket industry, titanium is recognized as a material of construction with many desirable properties for use with nitrogen tetroxide. The latter is well known as an economical and reliable storable liquid oxidizer for liquid-fueled rockets. Corrosionwise, titanium is unattacked by nitrogen tetroxide at temperatures up to 74°C. In contact with other strong oxidizers (red fuming nitric acid and oxygen) titanium is known to be impact sensitive and on occasion to react violently.

1.02 This project was initiated to determine the impact sensitivity of commercially pure titanium, titanium alloy 6Al-4V, and precipitation hardened 15-7 Mo stainless steel when exposed to liquid nitrogen tetroxide. Data are presented on these materials sheared, scraped, galled, and impacted by strikers and high velocity bullets. An explanation of the mechanism of the limited ignition resulting from impact is presented.

2. SUMMARY AND CONCLUSIONS

2.01 Commercially pure titanium and titanium 6Al-4V are mildly impact sensitive in liquid N_2O_4. The results are consistent with the theory that N_2O_4 is surface adsorbed on the pure titanium but not on the alloyed titanium. The adsorption process is essentially complete in one day. Commercially pure titanium soaked for 24 hours in N_2O_4 at 0°C will ignite about 50% of the time if struck with a flat-end pin having an energy of 60 - 70 ft-lbs. Titanium 6Al-4V, soaked or unsoaked, will ignite about 50% of the time if struck with a flat-end pin having an energy of 180 - 220 ft-lbs.

2.02 Ignition is rarely evidenced by sparks or noise and usually results in small fused areas on the impacted metal surface. Propagation of the reaction does not occur even though sufficient N_2O_4 is present to allow complete oxidation of the metal. The nitrogen evolved from the ignition reaction may slow the access of fresh oxidizer and permit time for the diffusion of heat to decrease temperatures below those required to continue vigorous oxidation.

2.03 Shear, scrape, gall, and high velocity impact data also indicate that titanium is not subject to propagating oxidation in N_2O_4 when impact ignited. The presence of grit on the titanium surface causes a marked decrease in the impact energy required for ignition. This is probably due to the increase in impact energy per unit area around a grit particle and a greater availability of bulk oxidizer at the instant of impact.

2.04 Precipitation hardened stainless steel, PH15-7 Mo (Armco condition RH-950) was not impact sensitive in liquid N_2O_4.

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3. MATERIALS

3.01 Nitrogen Tetroxide (N_2O_4) - A one-ton cylinder of commercial N_2O_4 was obtained from Nitrogen Division, Allied Chemical Corporation. The analysis and specification of this material are shown in Table 1.

Table 1 - N_2O_4 Analysis and Specification

	Analysis	Specification
N_2O_4 Assay, wt. %	99.84	99.5
H_2O Equivalent, wt. %	0.0363	0.1 max.
Cl as NOCl, wt. %	0.0048	0.08 max.
Non-Volatiles (ash), wt. %	0.0004	0.01 max.

3.02 Metals - The metals used in this study were commercially pure titanium (grades A-55 and 75-A), titanium alloy (grade 6Al-4V), and 15-7 Mo stainless steel precipitation hardened to Armco condition RH-950. The suppliers and certified analyses of these metals are shown in Table 3, Appendix I.

3.03 Preparation of Metal Specimens. Titanium and 15-7 Mo stainless steel specimens were cut from sheet stock using a water-cooled circular silicon carbide saw. Initially, 2 x 1/2 x 1/16-in. specimens were cut. Later, 3/4 x 3/4 x 1/16-in. specimens were cut to insure 100% contact with the 1/2 in. diameter impact strikers. Specimens were stamped with an identifying number, scoured with "Old Dutch Cleanser", rinsed with water, then in acetone, and dried. 15-7 Mo stainless steel specimens were scoured with "Old Dutch Cleanser", rinsed in water, then acetone, dried, and precipitation hardened to Armco condition RH-950. The specimens were again cleaned before use. Three-inch lengths of 3/4 in. ID titanium A-55 tubes for external impact tests were cut on a lathe and the ends machined smooth. These tubes were cleaned in the same manner as the flat titanium specimens.

4. APPARATUS AND PROCEDURES

4.01 Falling Hammer. The falling-hammer apparatus is illustrated in Figure 1. Hammers weighing 10, 21.33, and 25.7 pounds were used to impart up to 103, 220, and 254 foot-pounds of impact energy, respectively. Stainless steel (Type 304) and carbon steel firing pins, 0.5 inch diameter, with various shaped ends - flat, concave, and 90° point - were attached to the hammers to impart the impact. A clean specimen was placed in a tin-plated steel penetration cup (2-3/16 inch diameter x 1-3/8 inch deep - Emil Greiner Catalog No. 2041) and covered with 25 milliliters of liquid N_2O_4. Impact tests were made immediately, or the cup was closed with its companion top, placed in a covered Pyrex dish, and stored in a refrigerator at 0°C until used.

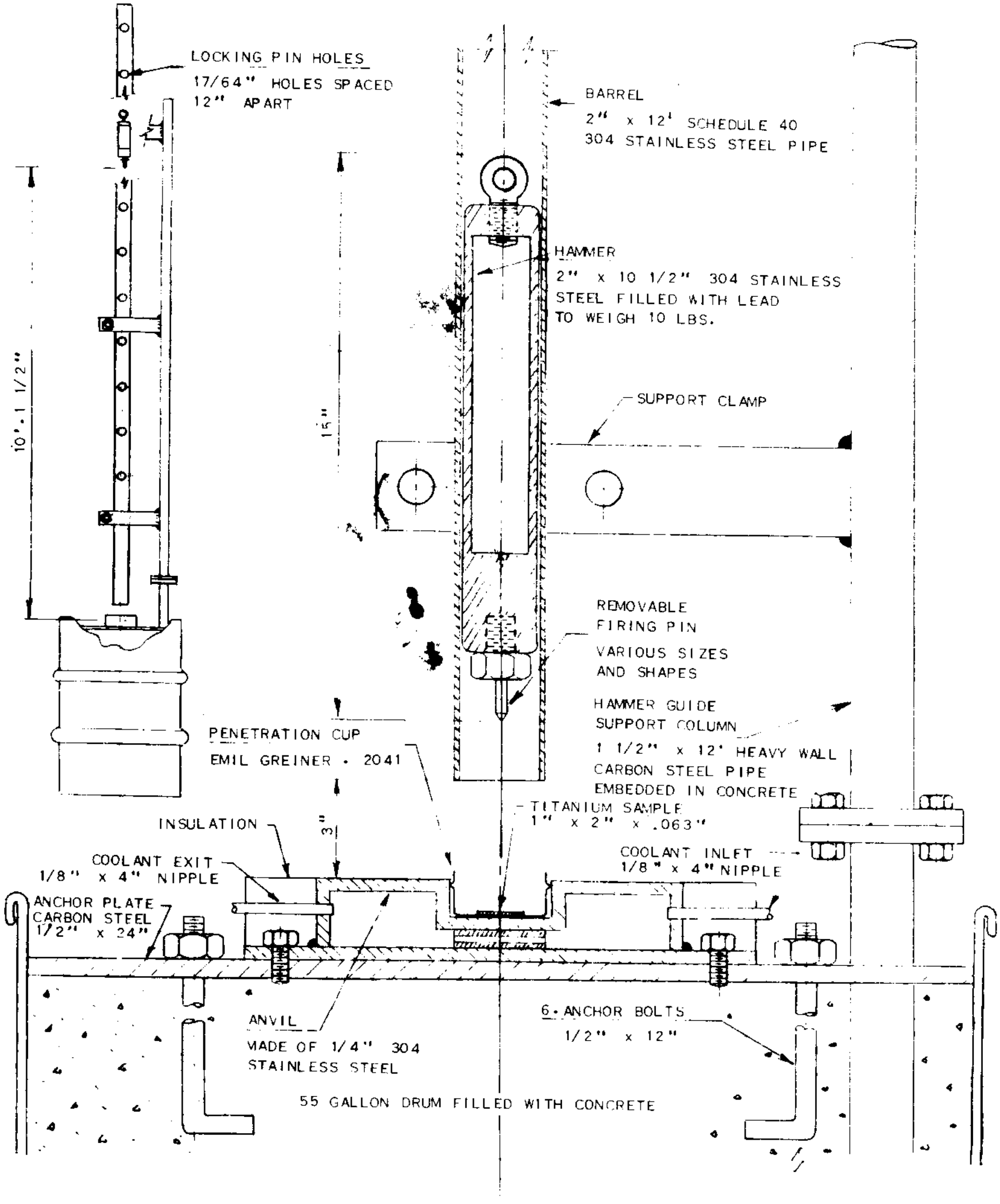


Fig. 1 - Falling-Hammer Apparatus

The cup containing the specimen and the N_2O_4 was placed in the anvil of the apparatus with the specimen centered directly under the firing pin. The hammer was placed at the desired height in the barrel and released by pulling a pin. Tests were made on the specimens with and without foreign matter added and at 2° and 22°C in an effort to establish an impact sensitivity threshold valve (energy required to ignite 50% of the specimens impacted).

4.02 Shear. This apparatus is shown in Figure 2. It was fabricated of Type 304 stainless steel and consisted of a combination cover and pin guide that held a 1/4-inch diameter shear pin perpendicular to the metal specimen. Two clamps held each specimen in position over a small anvil with a 0.281 inch diameter hole machined directly under the portion of the specimen covered by the end of the 1/4 inch diameter shear pin. Clean 1/2 x 1/2 inch specimens to be sheared were clamped to the anvil and placed on the bottom of a tin-plated steel penetration cup (2-3/16 inch diameter x 1-3/8 inch deep - Emil Greiner Catalog No. 2041) and covered with N_2O_4. The cup served as a container and as a support for the combination cover and pin guide. The entire assembly was placed in a refrigerator at 0°C until used. At the conclusion of the desired soaking period, the assembly was removed from the refrigerator and placed in the anvil of the falling-hammer apparatus. The hammer was placed at the desired height in the barrel and dropped on the 1/4 inch pin causing it to shear a 1/4 inch circle from the specimen.

4.03 External. Small titanium containers to be externally impacted were prepared from three inch sections of 3/4 inch inside diameter Grade A-55 titanium tubing. The tubes were mounted between two carbon steel flanges held against the tube ends by three 3/8 inch stud bolts. Teflon gaskets were used between the titanium and carbon steel flanges. One flange was fitted with a 1/8 inch pipe coupling and valve for loading purposes. High velocity external impact was provided by firing rifle bullets into the containers. For this purpose, high velocity, "Kopperklad", .22 caliber shorts (Winchester No. SS 22S) and Remington 30-06 caliber, 150 grain soft point bullets were used. Figure 3 is a dimensioned drawing of the container and Figure 4 shows the container mounted for test. The titanium containers were loaded with 18 milliliters of N_2O_4 which filled each to three-fourths of its capacity. These containers were kept outdoors (ambient temperature about 15 to 27°C) until externally impacted below the liquid level with a bullet fired from a shielded rifle at a distance of 21 feet. In addition to these tests, external impact tests were made on empty titanium containers for comparison. All containers were sawed in half lengthwise to allow close inspection of the inner surface.

4.04 Gall. Gall test were made by oscillating a 0.5 inch diameter flat-end pin loaded with a 10 or 58 pound dead weight on titanium specimens soaking in liquid N_2O_4. The falling-hammer apparatus was used to make these tests. The clean specimen was placed in a tin-plated steel penetration cup and

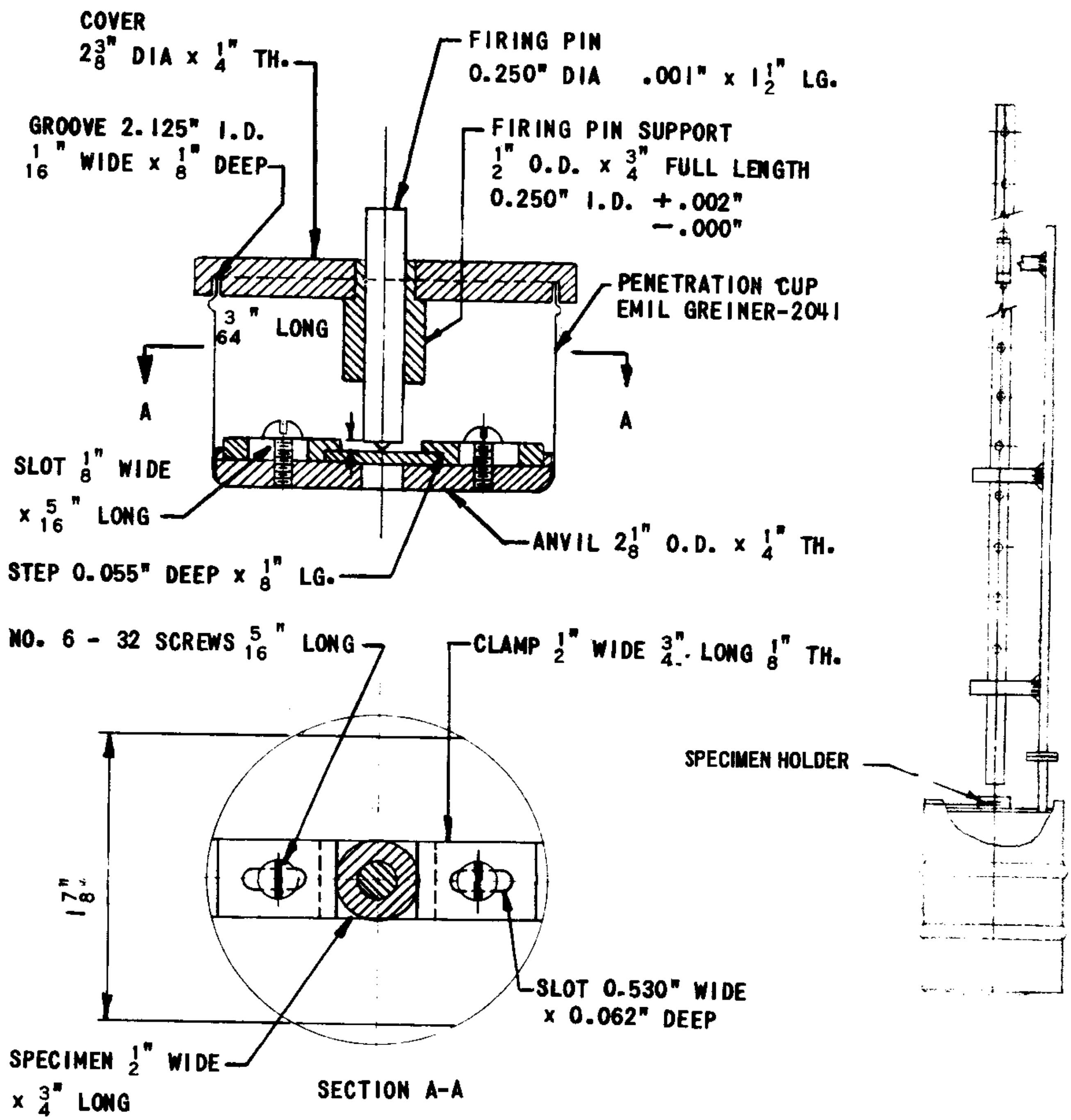


Fig. 2 - Shear Apparatus

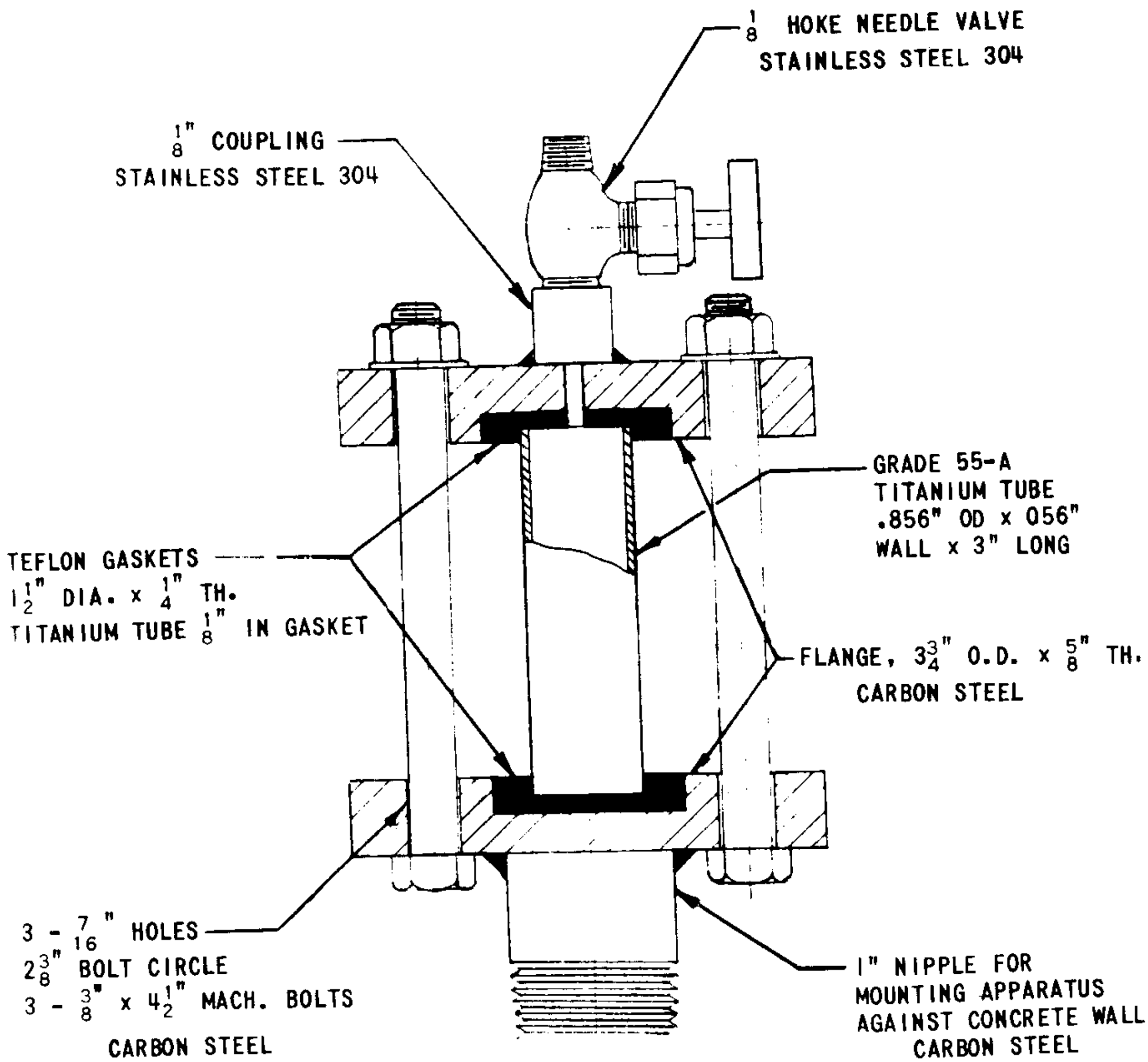


Fig. 3 - External Impact Container

Fig. 4 - External Impact Apparatus

covered with 35 milliliters of liquid N_2O_4. The cup was closed with its companion top, placed in a covered Pyrex dish, and stored in a refrigerator at 0^oC until used. The specimen was removed from the liquid N_2O_4 and immediately attached to the bottom of the anvil with stainless steel clamps. The specimen, which was still wet with N_2O_4, and the clamps were covered with a layer of the same N_2O_4 used for soaking the specimen. The dead weight with a flat-end pin, vertically supported in the barrel, was placed on the specimen and connected to the drive of a Hills-McCanna pump. The weight was oscillated through a 90^o arc for a desired period of time.

4.05 Scraping Gall. Scraping gall tests were made on titanium by manually pulling a sharp pointed tool bit across the specimens under N_2O_4. The falling-hammer apparatus and the procedure for soaking and anchoring the specimen described in Par. 4.04 were used for these tests. The specimens were gouged by rapidly pulling a tool bit, welded to the end of a 5-1/2 foot length of pipe, across the specimens. Metal was stripped from the specimens to a depth of 4 - 8 thousandths of an inch.

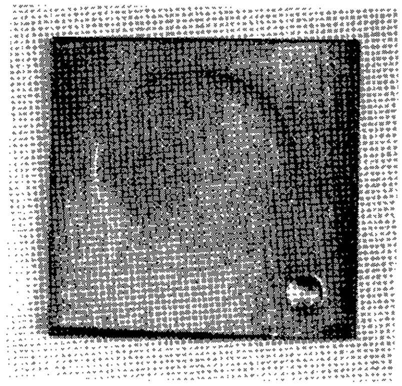
4.06 Impact Sensitivity of Stressed Titanium. Titanium specimens bent in a U-shape and stressed beyond the yield point were partially flattened and bolted in the slots of the anvils used in the shear tests as shown in Figure 2. Each specimen, held flat against the anvil with the side in tensile stress up, was placed in a tin-plated steel penetration cup and soaked in N_2O_4 for 24 hours at 0^oC. After soaking, each cup containing anvil, specimen, and N_2O_4 was placed in the anvil of the falling-hammer apparatus and the specimen was impacted with a 1/2 inch diameter flat-end pin.

4.07 Tensile Strength. Several specimens of titanium were soaked in liquid N_2O_4 and in its equilibrium gas at 15^o to 30^oC for two weeks and then tested for tensile strength. Stainless steel (304-L) containers 12 inches deep and 4 inches in diameter with a flanged head were used. Specimens were mounted on a carrier attached to the container head and glass was used to insulate the specimens from each other and from the stainless steel. Sufficient N_2O_4 was poured into one container to cover the titanium specimens completely. About 200 milliliters of N_2O_4 were poured in another container in which the titanium specimens were exposed to the gas above the liquid. After exposure the specimens were rinsed in water, dried, and their tensile strength and per cent elongation determined in a Model L Dillon tensile tester.

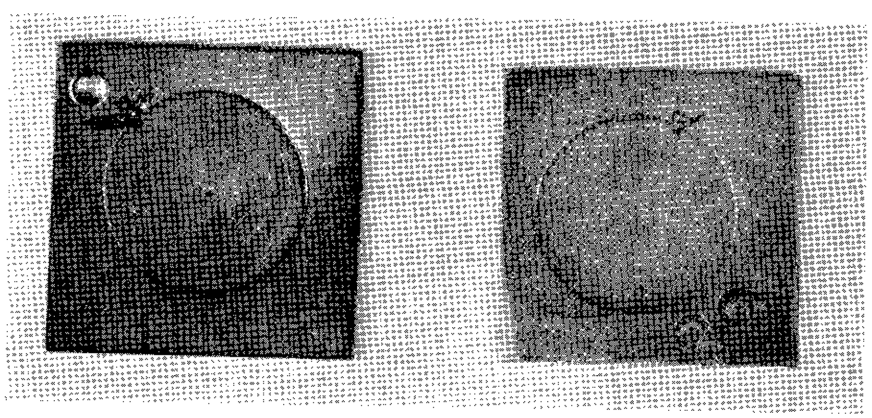
5. RESULTS

A. Impact Sensitivity of Commercially Pure Titanium and Titanium Alloy 6Al-4V

5.01 Surface ignition of titanium metals by impact in liquid N_2O_4 was observed under a variety of conditions. When ignition occurred as a result of flat impact, it was evidenced by small fused areas similar to those shown in Figures 5, 6, and 7. Ignition was seldom evidenced by sparks or noise. Propagation of the reaction did not occur even though sufficient N_2O_4 was present to completely oxidize the specimen.



A-55 Specimen
Not Ignited
By Impact
(2X)



A-55 Specimens
Ignited By
Impact (2X)

Fig. 5 - Impacted Titanium Specimens

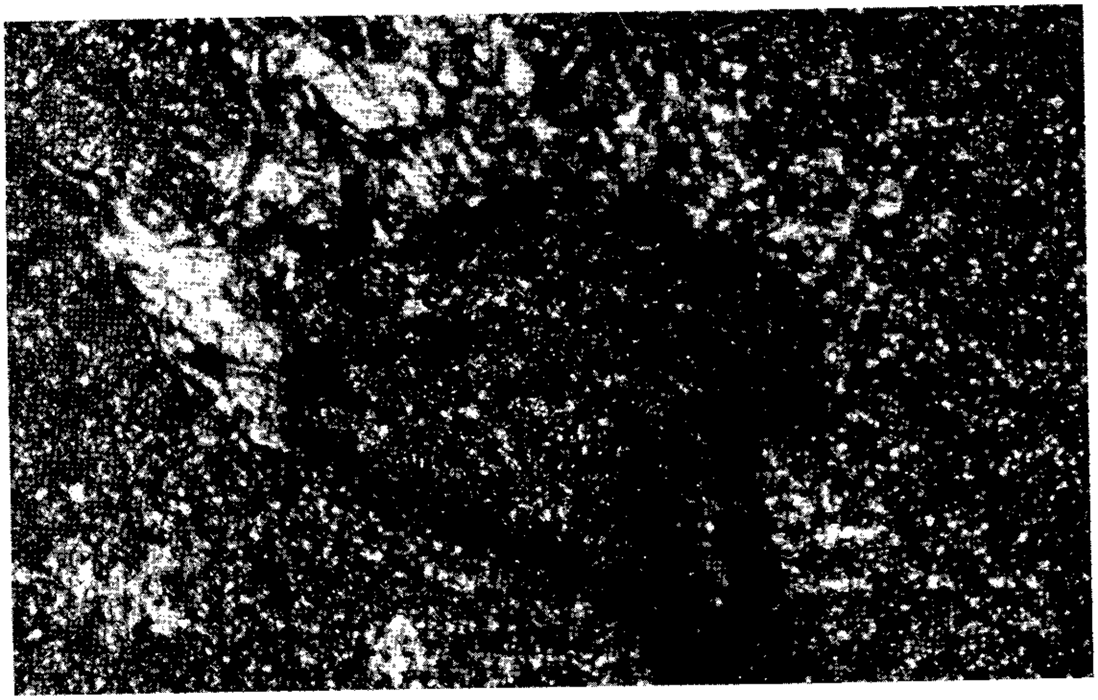


Fig. 6 - Typical Fused Area on Impacted Surface
(Approximately 150X)

Fig. 7 - Typical Fused Area on Impacted Surface
(Approximately 300X)

5.02 Commercially pure titanium soaked for 24 hours in N_2O_4 at 0°C had an impact sensitivity threshold value (energy required to ignite 50% of the specimens impacted) of 60-70 ft-lbs using a flat-end firing pin. Without soaking, and in N_2O_4 at 22°C, the threshold value was nearly 200 ft-lbs of impact energy. This threshold value decreased to 40 ft-lbs for one group of 75-A specimens after soaking 384 hours in N_2O_4 at 0°C; however, the value was 70 - 80 ft-lbs for another group of 75-A specimens soaked in N_2O_4 for 123 days at 0 - 10°C. These results indicate that sensitivity does not necessarily increase with soaking period longer than 24 hours. The presence of No. 240 grit Al_2O_3 on the titanium specimen in N_2O_4 lowered the threshold value to about 20 - 30 ft-lbs when using a flat-end pin. This value was obtained when the specimens were soaked in N_2O_4 for 24 hours at 0°C and in N_2O_4 at 22°C without soaking. Ignition did not occur on commercially pure titanium when 90° point (with and without Al_2O_3 on the specimens) firing pins were used. One attempt to ignite a soaked 75-A specimen by impact with a concave pin at the 103 ft-lb energy level was negative. A second attempt on another specimen at the 220 ft-lb level was positive only to a degree detectable by microscopic examination. Data are shown in Table 4, Appendix I.

5.03 Titanium alloy 6Al-4V soaked in N_2O_4 for 24 hours at 0°C had an impact sensitivity threshold value of 180 - 220 ft-lbs using a flat-end firing pin. The presence of No. 240 grit Al_2O_3 on this alloy in N_2O_4 at 22°C without soaking decreased the threshold value to 23 ft-lbs. Some ignitions occurred on 6Al-4V specimens soaked for 24 hours at 0°C when impacted in N_2O_4 with a 90° point pin having an energy of 113 ft-lbs. These ignitions were microscopically small and difficult to detect because of the overshadowing physical damage resulting from the impact. Data are shown in Table 5, Appendix I.

5.04 The hardness of the striking pins appeared to have no effect on the impact sensitivity within the range of 262 - 578 Brinell. Titanium 75-A soaked in N_2O_4 for 24 hours at 0°C ignited 50% of the time when impacted with 63 ft-lbs. of energy imparted by flat-end carbon steel pins heat treated to 495 and 578 Brinell hardness. These data compare closely with the 60 - 70 ft-lbs of energy required to ignite titanium 75-A with stainless steel pins having a Brinell hardness of 262. Indications are that hardening the firing pin above 262 Brinell does not decrease the amount of impact energy needed to ignite titanium. Data are shown in Table 4, Appendix I.

5.05 Commercially pure titanium soaked in liquid N_2O_4 for 72 hours at 0°C did not ignite when rubbed under the liquid with a 10-pound weight for 15 minutes, or with a 58-pound weight for one hour. The specimens were rubbed with weights having a flat-end (575 Brinell hardness), 0.5 inch diameter head oscillated through a 90° arc 142 times per minute. Titanium 6Al-4V showed no tendency to ignite after soaking in N_2O_4 for 24 hours at 0°C when rubbed under liquid N_2O_4 for one hour in the presence of No. 240 grit Al_2O_3 with a

58-pound weight. The specimen was rubbed with the weight oscillating through a 90° arc 142 times per minute.

5.06 Scrape-gall tests on commercially pure and 6Al-4V titanium soaked in N_2O_4 for 24 hours at 0°C were negative. A sharp tool bit was used to rapidly strip metal from each specimen to a depth of 4 - 8 thousandths of an inch under N_2O_4. The scraped specimens are shown in Figure 8.

5.07 All shear tests on titanium 75-A and 6Al-4V were positive. Ignition occurred at the shear but did not propagate. Data are in Tables 4 and 5, Appendix I.

5.08 Two titanium 75-A specimens, stressed beyond the yield point and held in stress during impact, did not ignite. Specimens were soaked in N_2O_4 for 24 hours at 0°C and impacted under N_2O_4 on the side in tensile stress. An impact energy of 53 - 63 ft-lbs was delivered by a 1/2 inch diameter flat-end pin.

5.09 External Impact. Small titanium A-55 containers, in which N_2O_4 was stored for 15 minutes, 24 hours, 72 hours, 168 hours, and 336 hours, were impacted with a .22 caliber bullet having a calculated energy of 79 ft-lbs. The impact caused a severe dent in each container about 1/4 inch deep with cracking on the interior surface. These cracks extended through to the exterior surface of the containers stored for 72, 168, and 336 hours. Close examination of the container interiors did not reveal any signs of ignition. Samples of the container walls are pictured in Figure 9.

5.10 An empty titanium tube suffered severe physical damage without ignition when externally impacted by a 30-06 caliber, 150 grain soft point bullet having a potential impact energy of 2884 ft-lbs. Two tubes containing N_2O_4, one for 15 minutes and the other for 96 hours, ignited over an area extending one inch from the point of impact. Ignition was accompanied by bright sparks and was evidenced by fused areas on the titanium surface. No propagation of the ignition was observed and neither container was damaged to a greater extent than the empty impacted container. The bullet passed through both walls of the container in the three shots fired. The container walls are shown in Figures 10 and 11.

5.11 Tensile Strength. Titanium 75-A and 6Al-4V soaked in liquid N_2O_4 and in its equilibrium gas at 15 to 30°C for two weeks showed no significant loss of tensile strength. Data are shown in Table 2.

TABLE 2

TENSILE STRENGTH OF TITANIUM SOAKED IN N_2O_4 FOR TWO WEEKS

Titanium Metal	Soaking Material	Ultimate Tensile Strength, psi	Elongation, % in 2 Inches
6Al-4V	None	148,100	14
6Al-4V	Liquid N_2O_4	148,100	14
6Al-4V	Gaseous N_2O_4 *	147,100	14
75-A	None	91,000	28
75-A	Liquid N_2O_4	92,300	28
75-A	Gaseous N_2O_4 *	92,300	28

* Specimens in vapor above liquid N_2O_4 in closed vessel at 15 to 30°C

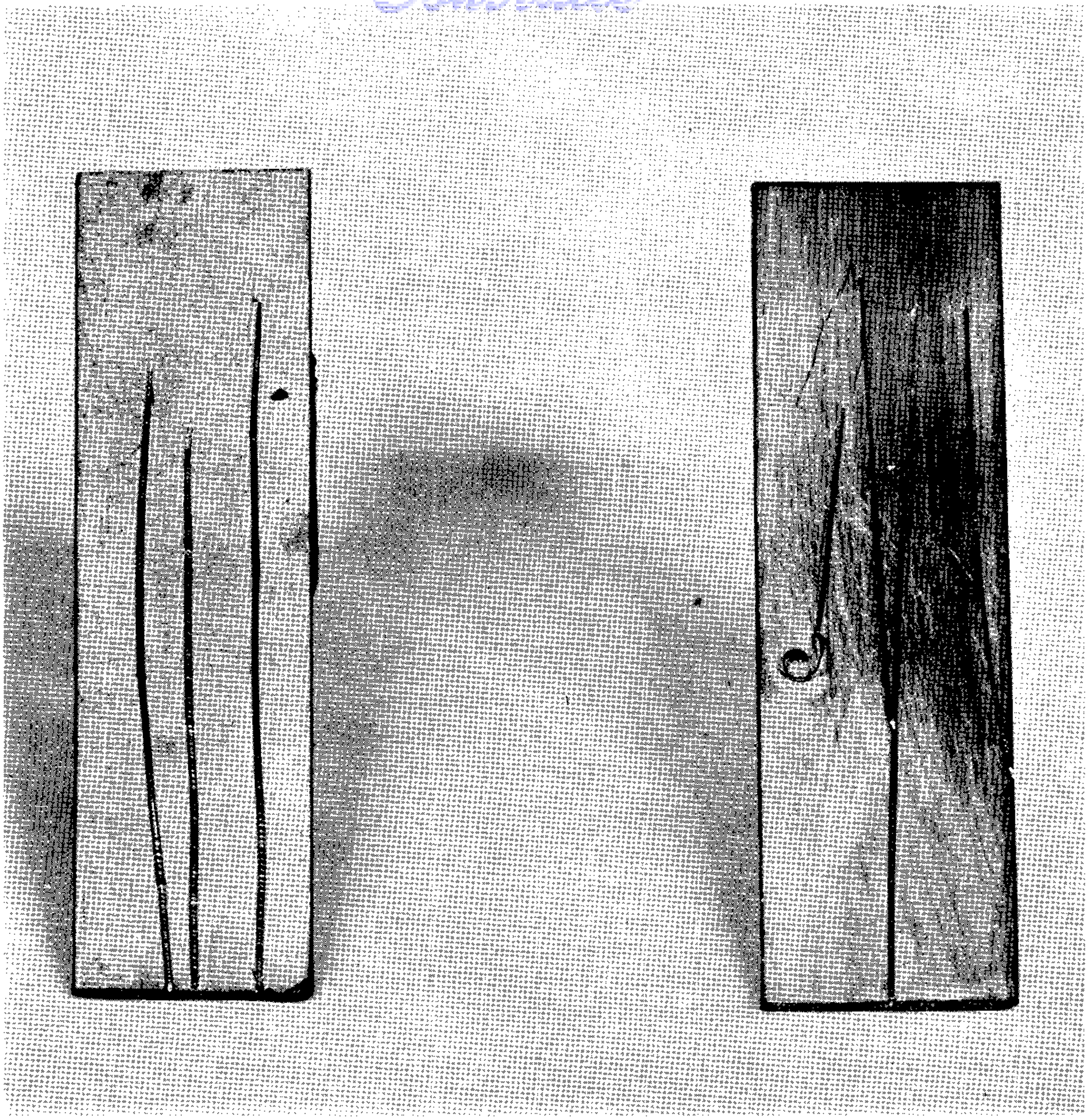
5.12 The impact sensitivity of titanium A-55 and 6Al-4V was not changed by soaking in wet N_2O_4 from the value obtained in dry N_2O_4. A threshold value of 73 ft-lbs was obtained by impacting A-55 specimens soaked at 0°C for 24 hours in N_2O_4 containing 0.5 wt % added water with a flat-end pin. The threshold value for titanium 6Al-4V was 197 ft-lbs.

5.13 Titanium soaked in liquid N_2O_4 for 1, 21, and 28 days at 50°C, dried, and stored in a dessicator for periods up to 3 weeks continued to show some residual sensitivity when rewetted with N_2O_4 and impacted with a flat-end firing pin. Data are shown in Table 6, Appendix I. The surface change indicated by these limited data did not appear permanent and seemed to diminish after about one week. Titanium A-55 soaked at 50° was not any more impact sensitive than that soaked at 0°C.

5.14 A number of 75-A and 6Al-4V specimens (1/16 x 1/16 x 1/2 inch) were heated on one end to a red heat just below the ignition temperature in air. The specimens did not ignite when plunged into gaseous or liquid N_2O_4. Similar specimens heated to ignition in air and plunged into gaseous or liquid N_2O_4 continued to burn. The burning in N_2O_4 (liquid or gas) was more rapid than in air but was not extremely vigorous or explosive.

B. Impact Sensitivity of PH15-7Mo Stainless Steel

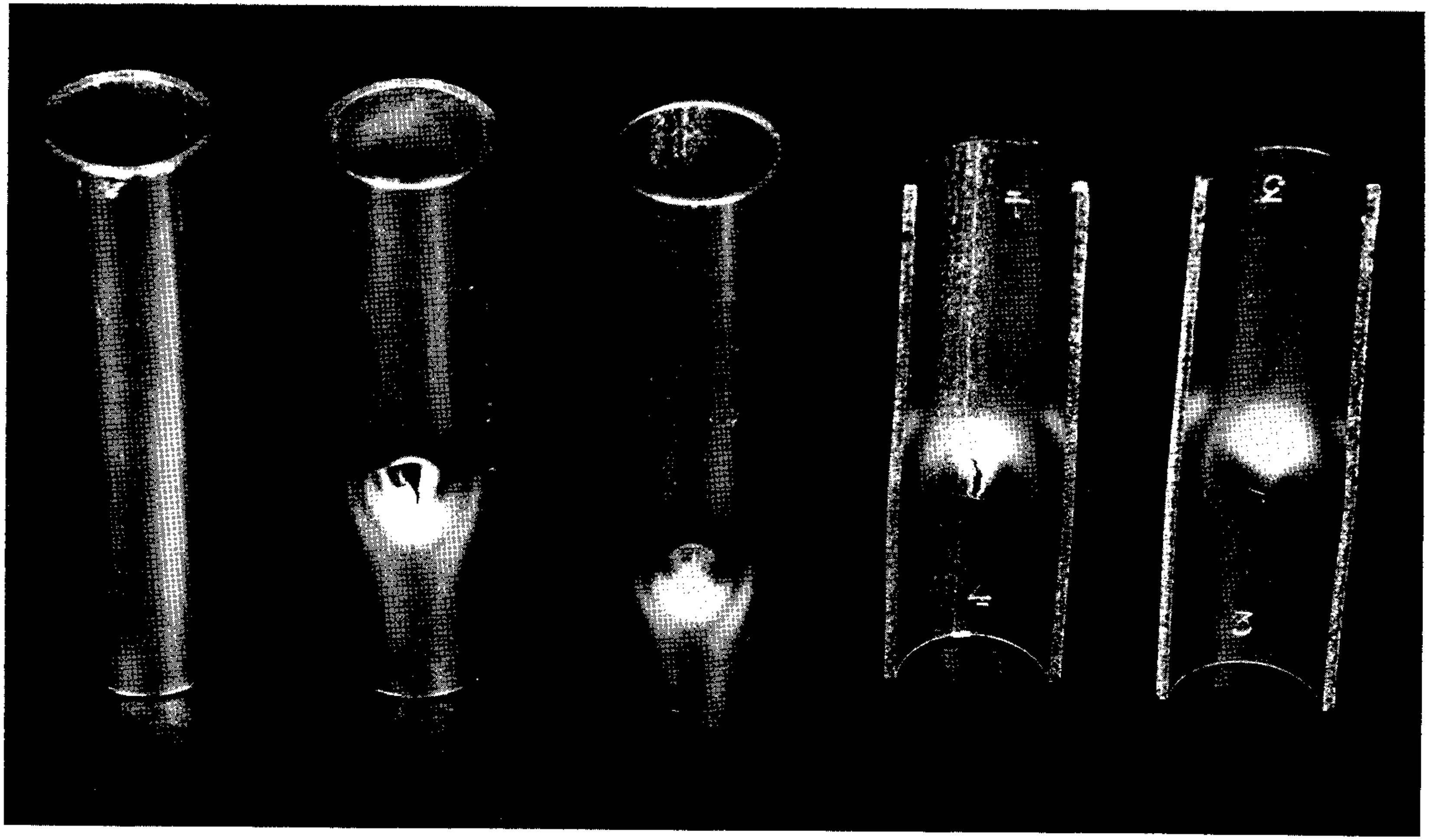
5.15 PH15-7Mo stainless steel (Armco condition RH-950) was not impact sensitive in N_2O_4. All tests using flat-end and pointed pins were negative even in the presence of No. 240 Al_2O_3 grit. Shear tests on this metal in



Titanium 75-A

Titanium 6Al-4V

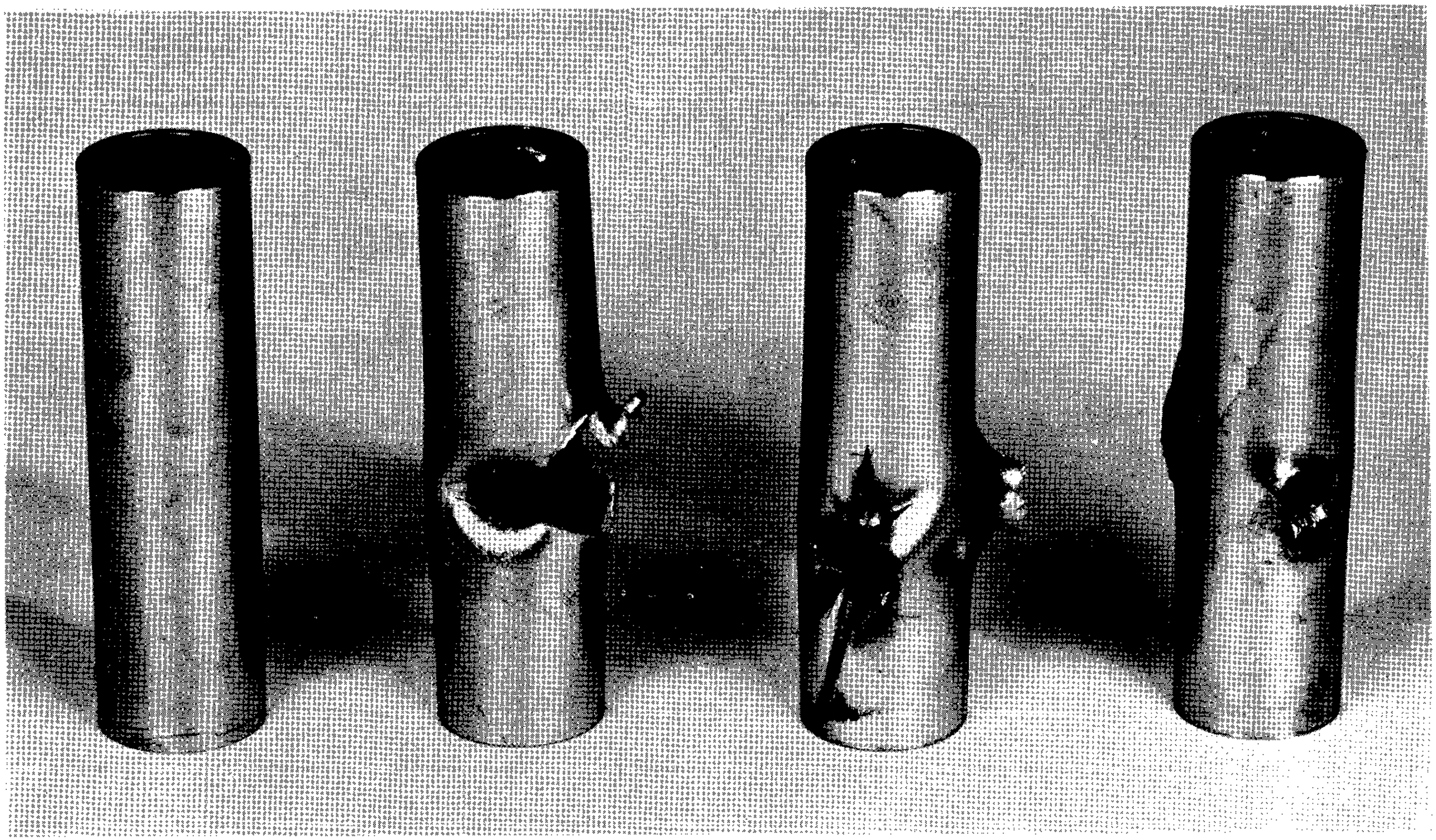
Fig. 8 - Titanium Specimens Scraped in N_2O_4



1 2 3 4 5

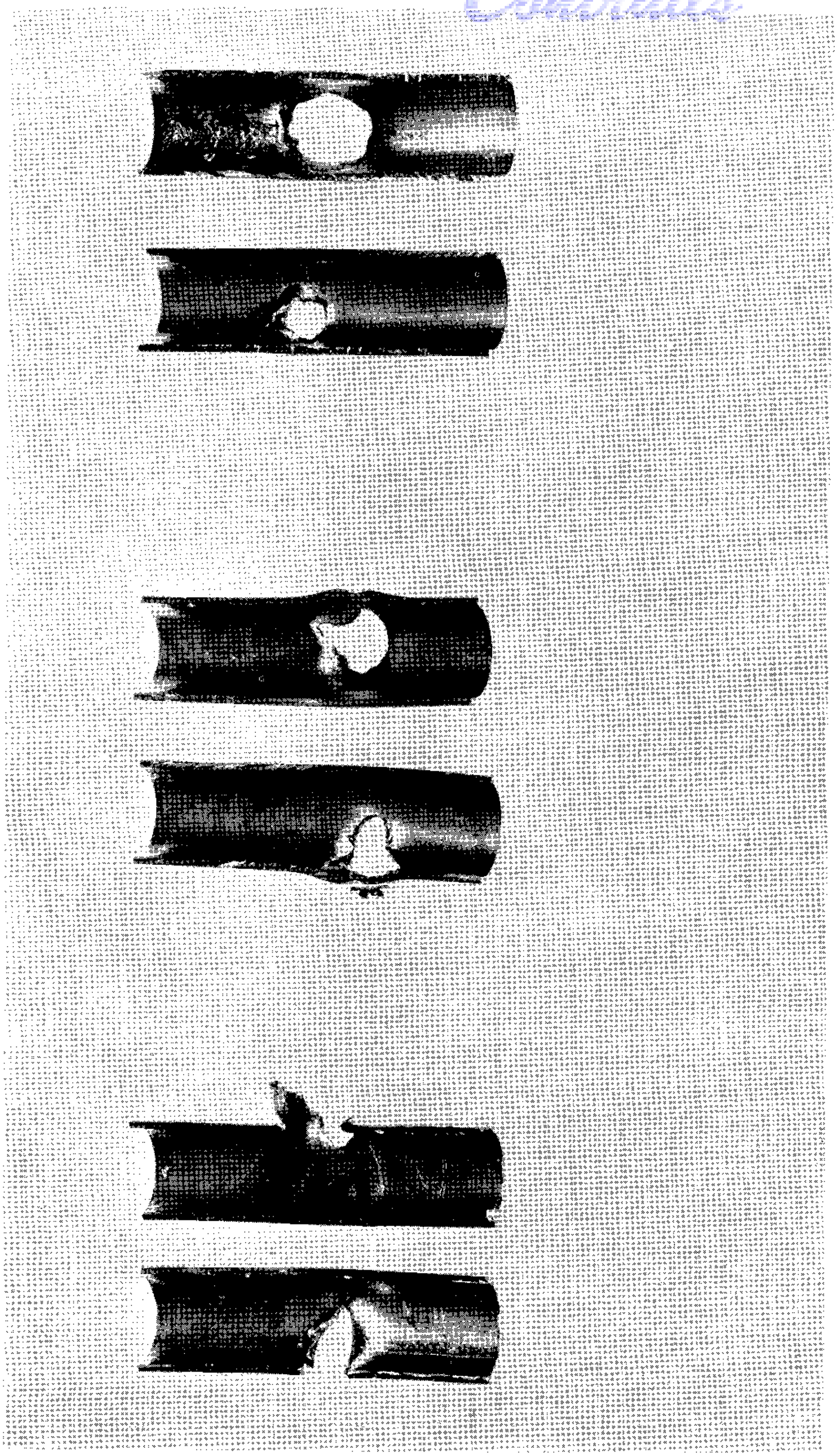
1. Titanium 55-A Tube
2. Titanium 55-A Tube Impacted With .22 Caliber Short
3. Titanium 55-A Tube Filled With N_2O_4 and Impacted After 1 Week's Storage
4. Internal Surface of Tube Filled With N_2O_4 and Impacted After 3 Days' Storage
5. Internal Surface of Tube Filled With N_2O_4 and Impacted After 1 day's Storage

Fig. 9 - Titanium A-55 Container Walls Impacted With .22 Caliber Bullet



Before Impact	Empty Before Impact	Filled With N_2O_4 and Impacted After 96 Hours	Filled With N_2O_4 and Impacted After 15 Minutes' Storage

Fig. 10 - Titanium A-55 Container Impacted With 30-06 Caliber Bullet(Exterior View)



Empty

Filled With N_2O_4
and Impacted
After 96 Hours'
Storage

Filled With N_2O_4
and Impacted
After 15 Minutes'
Storage

Fig. 11 - Titanium A-55 Container Impacted with 30-06 Caliber Bullet (Interior View)

N_2O_4 were negative. Data are shown in Table 7, Appendix I.

6. DISCUSSION OF RESULTS

6.01 The experimental data indicate that commercially pure titanium is impact sensitive in liquid N_2O_4. Ignition frequency as a function of impact energy is of the same order of magnitude as observed for flourine and oxygen in other laboratories (References 1 and 2) and less than fuming nitric acid (Reference 3). In no cases, however, has ignition been observed to cover more than a small fraction of the impact area as a surface fusion only. In this respect, it is more comparable to the results with flourine. There is a definite effect of time of exposure to the liquid over periods less than a day as about three times the impact energy is required for 50% ignition on samples impacted immediately after covering with N_2O_4 as compared with those exposed 24 hours to N_2O_4. Further exposure up to a few months has indicated no additional effects within the experimental accuracy. In this, it differs markedly from the fuming nitric acid results.

6.02 Adsorbed Film. The results from tests on commercially pure titanium are consistent with the reactive species being adsorbed N_2O_4 on the titanium surface. The adsorption process would appear to take appreciable time but is complete essentially in less than a day. Specimens stored in N_2O_4 for a period of days, dried and stored in a desiccator for up to three weeks, showed residual impact sensitivity when rewetted with N_2O_4. After a week or so ignition frequency decreases toward that of unexposed titanium. This corresponds to a gradual desorption of the sensitive adsorbed film. The adsorbed layer is complete at the vapor pressure of the liquid so no temperature effects are observed. The presence of alloying elements in the 6Al-4V titanium probably decreases the surface adsorption effects leaving the sensitivity close to that observed for unexposed titanium.

6.03 Corrosion Effects. With N_2O_4 even at elevated temperatures no corrosion within the experimental error was noted even with limited amounts of water in the N_2O_4 (Reference 4). In the fuming nitric acid system considerable corrosion occurs which results in increase in the surface area of titanium per unit of gross surface area as a powdery titanium coat is observed (Reference 3). This increase in surface adsorption area could account for the increased sensitivity with time of the fuming nitric acid system as compared to the N_2O_4 system where no increase in surface area occurs. The active surface adsorbed layer may well be the same in both systems. No intergranular corrosion and no change in tensile properties were noted on long exposure to N_2O_4 which tends to confirm that the active surface is the outer exposed surface. The fluorine and oxygen systems also show low corrosion and both show ignition frequency characteristics fairly similar to the N_2O_4 system.

6.04 Diffusion Effects. The N_2O_4 system and the flourine system both show very limited areas of ignition without continued combustion. In contrast, extended burned areas or continuing combustion may be observed in the oxygen system. The proposed explanation of Sterner and Singleton (Reference 1) also covers the N_2O_4 case. In the oxygen case, the rapid diffusion of the solid oxide into the molten titanium permits oxygen to reach the freshly exposed surface and continue the reaction. In the flourine case, the volatile flourides and in the N_2O_4 case the evolved nitrogen may slow down the access of fresh oxidizer and permit time for the diffusion of heat to drop temperatures below those required to continue ignition.

6.05 Grit. Tests in the drop weight tester show a marked decrease in impact required for a given value of the ignition frequency when hard grit is present. (For this reason, strikers were not ground with abrasive for a final polishing.) This is probably due to increase in impact energy per unit area around a grit particle and a greater availability of bulk oxidizer at the instant of impact. It is hard to picture as great an oxidizer compression where grit is present so this factor does not seem as important in this case as was proposed in the oxygen case (Reference 2).

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APPENDIX I

TABLE 3

Certified Mill Test of Metals

TABLES 4 THROUGH 7

Impact Sensitivity Test Data on Grades 75-A, A-55, and 6Al-4V Titanium and PH15-7Mo Stainless Steel

TABLE 3

CERTIFIED MILL TEST OF METALS

Material	Purchased From	Sheet No.	Heat No.	Chemical Composition	Test No.	Yield Pt.	Tensile Strength	Elon-gation	105° Bend Test Press Brake	Bend
Titanium - Grade A-55	Crucible Steel Company of America, Pittsburgh, Pa. Order No. DEV-30591	-	5446	H_2 .0041, C .04, N .02	-	77,700	88,700	28.7	-	2.0 T
Titanium - Grade A-55	Titanium Metals Co. of America, New York, New York Order No. HNR-25450	4-2	M-9082	C .026, Fe .28, N_2 .033, H_2 .004	A-2605 4L T	 82,500 87,400	 102,200 104,700	 21.0 22.0	 1.9 1.9	 - -
	Order No. DEV-30815	3-1 to 3-4	M-9562	.023, .10, .018, .004	A-3860 3L T	 75,900 81,000	 94,000 96,800	 25.0 25.0	 1.8 1.8	 - -
Titanium - Grade 6Al-4V	Titanium Metals Co. of America, New York, New York Order No. HNR-25450	12-1	M-8543	C .028, Fe .17, N_2 .011, Al 6.0, Va 4.1, H_2 .005	A-1364 12L T	 132,400 142,100	 142,900 148,500	 13.0 12.5	 3.6 3.9	 - -
PH15-7 Mo Stainless Steel	Armco Steel Corp. Middletown, Ohio	-	56254	C .075, Mn .60, P .021, S .006, Si .26, Cr 15.14, Ni 7.20, Mo 2.22	Condition RH-950	219,000	233,000	5-2"	Rockwell C 47	
Titanium Tube - Grade 55-A	Trent Tube Company East Troy, Wisconsin	-	-	Trentweld Type 55-A titanium pressure tubing, full finished (weld bead removed) annealed and descaled, manufactured to ASTM Specification A-269 except analysis. Tubing size - .856" OD x .056" wall x 9'-7" length.						

TABLE 4

IMPACT SENSITIVITY TEST DATA ON COMMERCIALLY PURE TITANIUM IN N_2O_4 (GRADES A-55 AND 75-A TITANIUM)

Soaked in N_2O_4 Hours	Soaked in N_2O_4 T°C	Type Firing Pin	N_2O_4 T°C	Foreign Matter On Sample	No. Samples Tested	Impact Ft-Lbs	No. Samples Impact Sensitive +	No. Samples Impact Sensitive -	Notes
-	-	Flat	22	None	1	220	1	0	
					6	201	5	1	
					3	179	0	3	
					1	137	0	1	
					10	113	1	9	
24	0	Flat	2	None	6	113	6	0	
					9	103	6	3	
					4	93	2	2	
					8	83	7	1	
					20	73	11	9	
					10	63	7	3	
					5	53	1	4	
48	2	Flat	2	None	1	113	0	1	
168	2				7	103	5	2	
336	2				1	100	1	0	
384	0				1	63	1	0	
384	0				5	53	4	1	
384	0				6	43	4	2	
24	2	90°	2	None	1	100	0	1	
93	2				1	100	0	1	
366	2				1	100	0	1	
24	0	Shear	2	None	1	113	1	0	
					1	83	1	0	
-	-	Shear	22	None	1	53	0	1	Did not shear
					2	83	1	1	Neg. sample did not shear
					1	73	0	1	Did not shear
24	0	Concave	2	None	1	103	0	1	
432	0		2		1	220	1	0	Ignition visible at 50X
	-	Flat	22	240 grit Al_2O_3	7	113	6	1	
				240 grit Al_2O_3	1	63	1	0	
				240 grit Al_2O_3	1	33	1	0	
				240 grit Al_2O_3	4	23	1	3	
				240 grit Al_2O_3	6	13	2	4	
24	0	Flat	2	Iron Filings	1	63	1	0	
				Iron Filings	1	33	1	0	
				SiC	1	63	1	0	
				SiC	1	13	1	0	
				SiC	1	13	0	1	
168	2	Flat	2	240 grit Al_2O_3	1	103	1	0	
264	2			240 grit Al_2O_3	1	103	1	0	
288	2			240 grit Al_2O_3	2	103	2	0	
336	2			Sand	3	103	3	0	
526	2			SiC	1	113	1	0	
24	0	90°	2	240 grit Al_2O_3	1	93	1	0}	Ignition visible with aid of microscope
24	0		2	240 grit Al_2O_3	1	63	1	0}	
24	0		2	240 grit Al_2O_3	1	43	0	1	

\+ Impact Sensitive
\- Not Impact Sensitive
Flat-End Firing Pin Is 0.5 in. Diameter. It is made of 304 stainless steel and has Brinell hardness of 262. This type pin used for all tests except where noted

TABLE 4 (Cont'd.)

IMPACT SENSITIVITY TEST DATA ON COMMERCIALLY PURE TITANIUM IN N_2O_4 (GRADES A-55 AND 75-A TITANIUM)

Soaked in N_2O_4 Hours	Soaked in N_2O_4 T°C	Type Firing Pin	N_2O_4 T°C	Foreign Matter On Sample	No. Samples Tested	Impact Ft-Lbs	No. Samples Impact Sensitive +	No. Samples Impact Sensitive -	Notes
24	0	Flat	2	None	1	33	0	1	Carbon steel firing pin - Brinell hardness of 495
					1	43	0	1	
					1	53	0	1	
					7	63	4	3	
24	0	Flat	2	None	1	33	0	1	Carbon steel firing pin - Brinell hardness of 578
					1	43	0	1	
					1	53	0	1	
					7	63	4	3	
72	0	Flat (Gall Test)	2	None	1	10	0	1	10 pound weight with flat-end pin (Brinell hardness 578) oscillated 142 times per minute for 15 minutes on sample under N_2O_4
72	0	Flat (Gall Test)	2	None	1	58	0	1	58 pound weight with flat-end pin (Brinell hardness 578) oscillated 142 times per minute for 60 minutes on sample under N_2O_4
24	0	Scrape	2	None	1	-	0	1	Sample scraped with sharp tool bit, gouging specimen to a depth of 0.004 - 0.008 in. with each pass of tool
24	0	Flat	2	None	1	53	0	1	Samples stressed beyond yield point and held in stress during impact.
					1	63	0	1	
24	0	Flat	2	None	6	73	3	3	N_2O_4 contained 0.5 weight % H_2O
2952	0	Flat	2	None	1	103	1	0	
					7	73	2	5	
					2	63	0	2	
					2	53	0	2	

\+ Impact Sensitive
\- Not Impact Sensitive

Flat-End Firing Pin Is 0.5 in. Diameter. It is made of 304 stainless steel and has Brinell hardness of 262. This type pin used for all tests except where noted.

TABLE 5

IMPACT SENSITIVITY TEST DATA ON GRADE 6Al-4V TITANIUM IN N_2O_4

Soaked in N_2O_4 Hours	Soaked in N_2O_4 T°C	Type Firing Pin	N_2O_4 T°C	Foreign Matter On Sample	No. Samples Tested	Impact Ft-Lbs	No. Samples Impact Sensitive +	No. Samples Impact Sensitive -	Notes
24	0	Flat	2	None	6	254	5	1	
24	0				20	220	8	12	
192	0				8	220	3	5	
24	0				10	201	3	7	
24	2				9	113	0	9	
24	2	90°	2	None	10	113	7	3	Ignitions visible with aid of microscope
-	-	Flat	22	240 grit Al_2O_3	5	43	5	0	
-	-		22	240 grit Al_2O_3	6	33	5	1	
-	-		22	240 grit Al_2O_3	4	23	2	2	
24	2		2	Sand	1	113	1	0	
24	0	Shear	2	None	4	53	4	0	
					1	43	1	0	
					1	33	1	0	
					1	23	0	1*	
24	0	Flat	2	240 grit Al_2O_3	1	58	0	1	58 pound weight with flat-end pin (Brinell hardness 578) oscillated 142 times per minute for 60 minutes on sample under N_2O_4
24	0	Scrape	2	None	1	-	0	1	Sample scraped with sharp tool bit, gouging specimen to a depth of 0.004 0.008 in. with each pass of tool
24	0	Flat	2	None	2	222	2	0	N_2O_4 contained 0.5 weight % H_2O
					4	197	2	2	
-	-	Flat	22	None	1	248	1	0	
					1	222	1	0	
					10	197	5	5	

* Did not shear
\+ Impact Sensitive
\- Not Impact Sensitive
Flat-End Firing Pin Is 0.5 In. Diameter

TABLE 6

RESIDUAL IMPACT SENSITIVITY OF TITANIUM A-55 AND 6Al-4V

Titanium	Days in N_2O_4 at 50°C	Days in Dry Storage After Soaking	Number of Specimens Impacted In N_2O_4	Impact Ft-Lbs	Impact Sensitive +	Impact Sensitive -
A-55	0	0	6	201	5	1
	0	0	3	180	0	3
	1	0	1	73	0	1
	1	0	3	103	0	3
	1	0	1	146	1	0
	1	0	1	171	1	0
	1	0	1	197	1	0
	1	21	2	73	0	2
	21	0	5	73	1	4
	21	3	1	83	1	0
	21	3	2	73	2	0
	21	15	2	73	0	2
	28	0	3	73	2	1
	28	0	1	63	0	1
	28	11	4	73	1	3
6Al-4V	0	0	10	197	5	5
	1	0	2	248	0	2
	1	21	4	171	2	2
	21	0	4	171	2	2
	21	4	4	171	3	1
	21	15	2	171	0	2
	28	0	1	197	1	0
	28	0	5	171	2	3
	28	11	1	171	0	1
	28	11	2	197	1	1

TABLE 7

IMPACT SENSITIVITY TEST DATA ON PH 15-7 Mo STAINLESS STEEL (ARMCO CONDITION RH-950) IN N_2O_4

Sample No.	Soaked In N_2O_4 Hrs.	Soaked In N_2O_4 T°C	Type Firing Pin	N_2O_4 T°C	Foreign Matter On Sample	Impact Energy, Ft-Lbs	Impact Sensitive	
1	24	0	90°	2	None	113	Negative	
2	24	0	Flat	2	Al_2O_3	113	Negative	
3	24	0	Flat	2	Al_2O_3	113	Negative	
4	24	0	Flat	2	Al_2O_3	113	Negative	
5	24	0	90°	2	None	113	Negative	
6	24	0	90°	2	None	113	Negative	
7	24	0	Flat	2	Al_2O_3	113	Negative	
8	24	0	90°	2	None	113	Negative	
9	24	0	Flat	2	Al_2O_3	113	Negative	
10	24	0	90°	2	None	113	Negative	
11								
12								
13	192	0	Flat	2	SiC	113	Negative	
14	24	0	Flat	2	Al_2O_3	220	Negative	
-	24	0	Shear	2	None	115	Negative	Sample sheared
-	24	0	Shear	2	None	73	Negative	Sample sheared
-	24	0	Shear	2	None	52	Negative	Sample sheared
-	24	0	Shear	2	None	30	Negative	Sample did not shear
-	24	0	Shear	2	None	30	Negative	Sample did not shear
-	24	0	Shear	2	None	52	Negative	Sample sheared

Flat-End Firing Pin Is 0.5 in. Diameter (0.197 sq in. face)
Pin For Shear Test Was 0.25 in. Diameter (0.049 sq in. face)

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