NFPA No.

481

PRODUCTION, PROCESSING, HANDLING AND STORAGE OF

TITANIUM 1972

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Official NFPA Definitions

Adopted Jan. 23, 1964: Revised Dec. 9, 1969. Where variances to these definitions are found, efforts to eliminate such conflicts are in process.

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Standard for the
Production, Processing, Handling and Storage of
Titanium

NFPA No. 481 — 1972

This standard was prepared by the NFPA Committee on Combustible Metals. It was initiated in 1955, tentatively adopted in 1957, and with certain revisions was finally adopted by the National Fire Protection Association in May 1958. Amendments were adopted in June 1959 and May 1961.

This 1972 edition is a complete revision of the 1961 edition, and was adopted at the May, 1972 Annual Meeting at Philadelphia, Pa.

Committee on Combustible Metals

Richard B. Smith, *Chairman*,
U. S. Atomic Energy Commission, Washington, D. C. 20545

S. E. Auck, Underwriters' Laboratories, Inc.
Victor O. Bohn, American Insurance Assn.
L. H. Bradley, National Automatic Sprinkler Fire Control Assn., Inc.
John Goudreau, Fire Equipment Manufacturers Assn.
H. J. Kautzman, Aluminum Company of America
Robert J. Krieger, Titanium Metals Corporation of America
R. B. Lawrence, Factory Mutual Research Corp.
Andrew S. Prokopovitsh, U. S. Bureau of Mines
A. F. Ratzer, Chemical Concentrates Corp.
Herman H. Spaeth, Insurance Services Office — Pacific Region
J. E. Troutman, Factory Insurance Assn.
T. H. Wright, Worthington, Ohio

Alternates.

R. P. Day, Factory Insurance Assn. (Alternate to J. E. Troutman)

SCOPE: To obtain and correlate information on fire hazards, methods of storage, handling and use, and fire protection of magnesium and other combustible metals, and to develop standards for the safe storage, handling and manufacture of these metals. Measures for the prevention or control of metal dust explosions are under the jurisdiction of the Committee on Dust Explosion Hazards.

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Standard for the

Production, Processing, Handling, and Storage of

TITANIUM

NFPA No. 481 — 1972

CHAPTER 1. INTRODUCTION

11. Purpose and Scope.

111. The purpose of this standard is to call attention to the fire and explosion hazards associated with the production, processing, fabrication, and storage of titanium; and to outline recommended methods of fire prevention, fire extinguishment, and safe personnel practices. Information and recommendations are based on the present state of the art developed through more than 20 years of commercial production and usage.

12. Definitions.

TITANIUM. A nontoxic metallic chemical element used principally in metallic alloy form.

SPONGE. A term used to identify the metal after it has been won from the ore but before it is melted into ingot. Sponge is a relatively finely divided form of the metal, nominally ½-inch or less screen size.

INGOT. The product of arc-melted sponge with or without the addition of other metallic alloying agents.

SWARF. Finely divided metal particles produced by sawing and cutting operations.

SHALL. Indicates a requirement.

SHOULD. Indicates a recommendation or that which is advised but not required.

APPROVED. Means acceptable to the authority having jurisdiction. The terms PROPERLY, SAFELY, SUITABLE and ADEQUATE shall be interpreted as conditions subject to determination by the authority having jurisdiction.

13. History of Titanium.

131. The element titanium was discovered by an English clergyman named William Gregor, who was an amateur chemist with an inquiring mind. In the makeup of the blacksands of Cornwall he discovered an element which at the time attracted no scientific interest. A few years later an Austrian, Klaproth, extracted the same element from an ore called rutile. He named it Titanic Earth — for the mythical Titans, sons of earth.

132. Titanium was first produced in the United States about 1910 by Doctor M. A. Hunter of Rensselaer Polytechnic Institute. The basic process for the production of titanium metals today was developed many years ago by Wilhelm Kroll, a native of Luxembourg. His patent was vested by the U.S. Government during World War II and experimentation continued by the U.S. Bureau of Mines. In 1946, the Bureau of Mines announced the operation of a pilot plant producing titanium in batch lots by the Kroll process.

133. Commercial production started in 1948 in a plant having a capacity of 100 pounds a day, or less than 20 tons a year. By 1951, the fulfillment of the needs of the military forces had brought about tremendous strides in the titanium industry. Large scale commercial production had become a reality.

134. Titanium-bearing ores are plentiful and widely scattered about the world, including the United States. The principal ores are rutile and ilmenite. At present rutile is the simpler and most desirable ore for titanium recovery. It is, however, in the shortest supply, coming primarily from deposits in Florida and Australia. It is generally recognized that, in time, the greatest tonnage of titanium metal may be processed from ilmenite ore. The largest ilmenite mine in the United States is located in Tahawus, New York.

135. Titanium sponge is currently produced in the United States, Japan, England, and the Soviet Union. Three basic processes have been developed for commercially refining titanium from rutile. The most widely used processes utilize magnesium or sodium to reduce titanium tetrachloride. An electrolytic process has also been proven to be practical.

136. Titanium ingot is produced by arc-melting a consumable electrode of compacted sponge, or sponge and alloy, into a cooled copper mold under a low vacuum or an atmosphere of inert gas.

14. Properties of Titanium.

141. Silver-grey titanium is classified as a light metal, being 60 percent heavier than aluminum, but only 56 percent as heavy as alloy steel. Titanium-base alloys are stronger than aluminum alloys and most alloy steels. Titanium has excellent ductility (easily drawn-out or hammered thin).

142. Titanium alloys are superior to all the usual engineering metals and alloys in strength-weight (strength divided by density or weight) ratio. Its fatigue resistance (ability to resist repeated flexures) is above that of heat-treated alloy steels and far above that of nonferrous metals. Titanium alloys are harder than aluminum and approach the high-alloyed steels. Surface hardness comparable to nitrided steel is obtainable.

143. Titanium is highly corrosion resistant, being greatly superior to aluminum, considerably better than many specialty steels, and unique among the common engineering metals in its immunity to salt water and marine atmosphere.

a. Titanium is the only known structural metal highly resistant to attack by simultaneous exposure to sea water and air.

144. As for impact resistance (the capacity to withstand shock) titanium is superior to aluminum, and some titanium alloys have impact resistance approaching that of heat-treated steels.

145. Titanium loses strength and becomes embrittled on continued exposure to air at temperatures above 1000°F.

146. Titanium has an atomic weight of 47.90, specific gravity of 4.5, and a melting point of 3140° F. Information on available grades, tensile strength, yield strength, ductility, hardness, fatigue properties, corrosion resistance and other mechanical and chemical characteristics is available from titanium producers, the U.S. Bureau of Mines, et al.

a. Normal compositions of some widely used commercial alloys are as follows:

Titanium 90%, Aluminum 6%, Vanadium 4%

Titanium 92.5%, Aluminum 5%, Tin 2.5%

Titanium 90%, Aluminum 8%, Molybdenum 1%, Vanadium 1%

Titanium 86%, Aluminum 6%, Vanadium 6%, Tin 2%

Titanium 92%, Manganese 8%

147. Titanium presents some fire hazards during production of the raw sponge, melting of the sponge, casting, machine operations that produce fine turnings or chips, powder production and the handling and disposal of scrap which contains chips or fines. However, because of its high temperature properties in the solid form, titanium sheet is extensively used for fire walls in jet aircraft and spacecraft.

148. In molten form, titanium either dissolves or is contaminated by every known refractory. Slight contaminations apparently have little effect on the flammable characteristics of chips, turnings or powder produced in machining operations, but may have an important bearing on ignition and explosion hazards associated with acid or salt bath treatments.

145. Titanium combines readily with oxygen, nitrogen and hydrogen at temperatures considerably below its melting point. Freshly exposed surfaces have a tendency to quickly form an adherent oxide coating evidenced by discoloration.

15. Tests for Titanium.

151. Two relatively simple methods are available to assist in separating solid titanium components from other metals. It is very important that all other metals be removed from titanium scrap before recycling.

a. Spark Test. Distinctive sparks are thrown off when a piece of titanium is held against a grinding wheel. The white lines traced by the flying sparks end with a burst that produces several brilliant white rays or branches.

b. Glass Test. The softer grades of titanium and titanium alloys are able to wet glass and can be identified by rubbing a moistened piece of the metal on a piece of glass. If the metal is relatively soft titanium it will leave distinctive grey-white marks on the glass.

152. A portable metal spectroscope will better serve the purpose when attempting to identify titanium scrap by grade.

16. Applications.

161. Titanium has many uses. However, production is still being largely consumed by commercial and military aircraft manufacturers for use in jet engines, airframes and outer skin covering on sonic and supersonic planes. Titanium is being used in space vehicles and communication satellites.

162. Other military uses include armour plate, electrical components, pontoons, cables, structural braces, fire walls, personnel helmets and protective vests. A nonmilitary submarine hull is currently being fabricated from titanium.

163. Titanium's virtually complete immunity to atmospheric and salt water corrosion and such media as wet chlorine, nitric acid and most oxidizing chemicals makes it attractive for chemical plant applications such as heat exchangers, dryers, mixers and many other uses.

164. Specially prepared, very finely divided titanium powders find limited application in powder metallurgy and other relatively small scale uses.

17. Combustibility and Explosibility.

171. In tests at the Bureau of Mines laboratories, with titanium powders having a particle size of less than 200 mesh, ignition of dust clouds in air were obtained at temperatures ranging from 330° C to 590° C (626° F to 1094° F) while ignitions of dust layers occurred at 380° C to 510° C (720° F to 950° F). In some cases, dust clouds ignited at lower temperatures than static layers of the same dust. See Bureau of Mines Report of Investigation, Nos. 3722 and 4835.*

172. Titanium fines, nominally under +48 mesh, a by-product of sponge production and handling, and coarser particles such as the swarf, produced in sawing and grinding operations, can be ignited by a spark.

173. Tests conducted at Underwriters' Laboratories and reported on Data Cards 511 and 512** showed that dry ductile titanium in the form of thin chips and fine turnings could be ignited with a match. Normal size machine chips and turnings ignited and burned when heated in the flame of a Bunsen or blast burner. When ignited, titanium sponge or coarse turnings burn slowly with the release of great heat, although a sponge fire may spread rather rapidly immediately after ignition.

174. Heavy castings or ingots may give some indication of burning when being cut with an oxyacetylene torch, but when sufficient surface is available to permit radiation cooling below the critical temperature, the burning ceases when the torch is removed.

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175. Combustion of titanium can occur in atmospheres other than air. One of the fine powder samples that ignited in air as a cloud at 480° C (900° F), and as a layer at 470° C (860° F) could also be ignited as a layer in pure carbon dioxide at 680° C (1260° F). At red heat (1300° F) titanium decomposes steam to free hydrogen and above 1475° F titanium burns readily and vigorously in atmospheres of pure nitrogen.

18. Special Hazards.

181. Resistance to corrosion has been stressed as one of the outstanding properties of titanium. It does not rust, is not affected by sea water and resists the attack of many corrosive acids. Experiences have indicated, however, that extreme care should be taken in attempting to use titanium metal or powder in red fuming nitric acid. While no difficulty has been reported in using normal nitric acid, explosions have occurred in laboratory tests with titanium and red fuming nitric acid. Completely satisfactory explanations of these very rare explosions have not been developed. It is believed that the strength of the acid is an important factor and that some pyrophoric material is produced which, when disturbed, releases a large amount of heat permitting rapid oxidation of the metal. Potentially hazardous reactions between titanium and various chemicals are listed in NFPA No. 491M*.

182. Spontaneous Combustion.

a. Spontaneous combustion has occurred in fine, water-soluble oil-coated titanium chips and swarf. Such fires, while probably due primarily to the presence of oil and certain contaminants, are very difficult to control, and special precautions shall be taken to have all fine scrap and, particularly, oil-covered material removed from the plant and stored where any fire that may occur can be segregated and prevented from communicating with other combustible material.

b. Dry titanium fines collected in cyclones have on occasion ignited spontaneously when allowed to drop freely through the air. Also, sump fines will often ignite when they are dried.

c. During the early stages of the development of the titanium industry, thin titanium sheets were reported to have ignited spontaneously as they were being removed from a descaling bath of sodium hydride. However, the use of a potassium hydride solution in recent years has eliminated this problem.

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183. Molten metal and water.

a. Like any other metal in the high temperature molten state, titanium can cause a violently destructive explosion if water is present in any mold, pit or depression into which the fluid metal is poured or spilled. Under such circumstances, severe damage may be caused by ordinary steam pressure, an exothermic chemical reaction or a low order hydrogen-air explosion.

b. In the 1950's several violent explosions occurred in titanium consumable electrode melting furnaces, when water was allowed to enter the furnace through a crucible failure resulting from loss of cooling water flow and severe arc through, or melting through, the bottom. A committee composed of industry representatives prepared a set of general recommendations on design features for titanium and zirconium production melting furnaces to improve the safety of the process. Their recommendations, given consideration in the development of this standard, have been published by the Defense Metals Information Center, Battelle Memorial Institute, under the title General Recommendations on Design Features for Titanium and Zirconium Production Melting Furnaces.*

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CHAPTER 2. SPONGE PRODUCTION

21. Processes.

211. Titanium tetrachloride ($TiCl_4$), which is made from rutile (approximately 95 percent TiO_2) by high-temperature reaction with chlorine gas in the presence of a reducing agent, usually carbon, is currently used for all commercial production of titanium metal. There are three basic commercially used processes for reducing titanium from tetrachloride; the Kroll-Bureau of Mines process, utilizing magnesium metal as a reducing agent, the sodium process which utilizes liquid sodium as a reducing agent, and the electrolytic process. The result of these processes is a product referred to as titanium sponge.

212. In the Kroll-Bureau of Mines process purified titanium tetrachloride is fed into a steel reaction chamber containing molten magnesium. The reduction takes place under an inert gas blanket of argon or helium when the reactor is heated between 700° C and 900° C, producing titanium sponge (so-called because the mass found in the reactor has a spongy appearance) and magnesium chloride. The magnesium chloride is drawn off in the molten state for storage or reprocessing to magnesium and chlorine. After cooling, the sponge mass is bored from the reactor vessel in a dry room, and crushed into a useable size. A process of acid leaching or vacuum distillation is required to remove a residue of magnesium and magnesium chloride. A modified version of the Kroll process involves vacuum distillation in the reaction vessel before removal of the sponge for shearing or crushing. A dry room is not necessary. A detailed and illustrated description of the reduction process and equipment is given in Bureau of Mines Report of Investigations No. 4879.* The commercial production of titanium metal by the magnesium reduction process is also described by R. L. Powell in Chemical Engineering Progress, November, 1954, pages 578 through 581.

213. In the sodium reduction process, liquefied sodium is used as the reducing agent. The reaction vessel is heated to approximately 1000° C. There is no withdrawing of a by-product during the reduction cycle, such as is necessary with the magnesium process. After completion of the reduction process, the reactor contains a solid mixture of titanium sponge and sodium chloride. The mixture is referred to as "spalt". After a selected cooling period the mixture is bored from the reaction vessel. A dry room is not required. After

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removal from the reaction vessel the spalt is vacuum dried. The commercial production of titanium metal by the sodium reduction process is described by T. Peter Forbath. "Sodium Reduction Route Yields Titanium," Chemical Engineering, March, 1958, pages 124-127.

214. In the electrolytic process, tetrachloride is fed into a cell containing a molten salt bath, such as sodium chloride, where it is reduced to crystalline metal by fused salt electrolysis. The crystalline mass must be crushed, leached, and dried after removal from the cell. This process has been proven to be commercially feasible but has not yet been used to produce significant amounts of sponge.

215. The titanium sponge fire risk is affected by the process. Both the sodium reduction process and the electrolytic process produce a more crystalline sponge which has less tendency to be pyrophoric than the magnesium reduced sponge. Titanium fines resulting from crushing magnesium reduced sponge also tend to be more pyrophoric than fines produced from sodium reduced or electrolytic sponge.

22. Processing Equipment.

221. Reduction furnaces should be air-cooled. Furnaces shall be kept dry and free of iron scale and spillage.

222. Fuel supply lines to gas- or oil-fired furnaces shall have control valves at an easily accessible location remote from the reduction furnaces.

223. Fans handling combustible dust or gas and air mixtures shall be constructed in accordance with NFPA Standard No. 91.*

224. All electrically operated or controlled processing equipment shall be installed in accordance with the National Electrical Code, NFPA No. 70.*

225. All processing equipment shall be installed to meet local, state, and federal noise, air and water pollution standards and safety requirements.

23. Plant Construction.

231. Buildings housing reduction furnaces, boring and crushing facilities, and magnesium refining operations shall be of noncombustible construction. Provision for explosion venting should take into consideration Bureau of Mines findings.

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232. All buildings shall have adequate ventilation.

233. Building exits shall meet the requirements of the Life Safety Code, NFPA No. 101.*

234. Floors in reduction, boring and crushing buildings shall be noncombustible, preferably of concrete, brick, or steel plates. Floors should be slightly crowned to prevent any accumulation of water in the vicinity of reduction furnaces.

24. Storage of Raw Materials.

241. Magnesium ingots for use in the Kroll process should be stored in accordance with NFPA No. 48.*

242. Chlorine is a toxic nonflammable gas. For fire and explosion hazards refer to NFPA No. 49.* It has both a liquid and gas phase in pressurized containers. All containers used in the transportation of chlorine, as well as the means of transportation, are controlled by Department of Transportation regulations. Chlorine containers should be handled and stored in accordance with recommendations of the Chlorine Institute, Inc., as published in the Chlorine Manual.**

243. Titanium tetrachloride ($TiCl_4$) containers should be stored in a cool, well ventilated dry place away from areas of acute fire hazards. Containers should be plainly labeled and tightly sealed until used. The shipment of titanium tetrachloride is covered in the hazardous materials regulations of the Department of Transportation.†

244. Acids in quantity should be stored in specially designed tank farms provided with personnel safety equipment. For fire and explosion hazards, and life hazards, refer to NFPA No. 49, Hazardous Chemicals Data.*

25. Dust Collection.

251. Magnesium chloride dust resulting from the boring and crushing of titanium sponge produced by the Kroll process should be collected in bag houses or removed by wet scrubbers. Since magnesium chloride is hygroscopic, bag houses should be kept warm and dry.

252. Cyclone dry collectors are recommended where sponge is continuously fed from dryers. Collectors shall be emptied daily.

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253. Regulations limiting the amount and/or opacity of particulate matter that can be emitted into the atmosphere are being, or have been, adopted by federal, state and, in some areas, local agencies. Installation of dust collecting equipment generally must be made in accordance with such regulations, particularly Public Law 90-148, the Air Quality Act of 1967.

26. Fire Prevention.

261. Because of the great affinity of titanium and magnesium for oxygen, particularly at elevated temperatures, as well as the potential for magnesium and/or sodium ignition in air, the reduction process shall be carried on in an enclosed oxygen-free container, both for reasons of quality control and fire prevention. See NFPA No. 69.*

262. To insure the effectiveness of the inert gas system in reactor rooms, precautions shall be taken to always have available a sufficient supply of inert gas to meet anticipated needs and a reserve supply for emergency use. All pipes, fittings, valves, etc., in the inert gas dispensing or distributing system should be checked to insure an uninterrupted flow of gas to the reactors or elsewhere as needed.

263. Since titanium fines (normally everything under +48 mesh) can be ignited with a small spark, accumulations of fines in crushing and drying systems must be prevented. Sponge discharged from dryers shall be collected in increments no larger than tote bins or 55-gallon drums. The collection area shall be well-vented and free of combustible material. A fork truck or other suitable hauling vehicle shall be available at all times for quick removal of burning containers. Sponge containers shall never be stored in this area.

264. All systems shall be thoroughly cleaned of titanium fines and sponge before attempting maintenance work. All equipment and adjacent areas shall be washed down with water before proceeding with any welding or cutting on the processing equipment or in the immediate area.

265. All containers used to receive molten metal, molten magnesium, molten magnesium chloride, or liquid sodium must be thoroughly cleaned and dried before using. All pieces of magnesium metal must be clean and dry when charged to reactors.

266. Good housekeeping is essential. Supplies shall be stored in an orderly manner with properly maintained aisles to permit regular inspection and segregation of incompatible materials. Supplies of material in the reactor rooms and drying rooms shall be limited to amounts needed for normal operation.

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267. Ordinary combustible material such as paper, wood, cartons, packing materials etc., shall not be stored or allowed to accumulate near furnaces, dryers, or other ready sources of ignition.

268. Smoking shall be prohibited in all areas where titanium sponge is bored, crushed, dried or stored, and in all areas where titanium fines are produced or stored.. Areas shall be clearly posted.

269. Boring, crushing and drying equipment shall be properly earth grounded to dissipate static charge build-up. See NFPA No. 77.*

27. Fire Protection.

271. Water and other liquids have proven ineffective in extinguishing titanium sponge fires. Streams of water intensify the burning by feeding oxygen to the fire. There is also the possibility of creating a steam or hydrogen explosion, particularly if large amounts of sponge are involved. The great affinity of high-temperature titanium for oxygen will free considerable hydrogen which may reach explosive concentrations in tightly confined spaces. Entrapment of water under any burning or hot molten metal may result in a steam explosion.

272. Experience has shown that dry sodium chloride is one of the most effective chemicals for containing titanium sponge or fines fires. Another is a nonmetallic flux compound consisting largely of potassium chloride, magnesium chloride and calcium fluoride. Commercial dry fire extinguishment powders listed by Underwriters' Laboratories, Inc. are also effective. Covering the fire completely reduces the accessible oxygen supply, thereby slowing the burning rate so that eventual extinguishment may occur.

273. Automatic sprinkler systems are recommended for office buildings, storehouses and repair shops. However, sprinkler systems are not recommended for installation over titanium sponge production, crushing, leaching, drying, chlorinator, magnesium refinery, sodium storage, blending facilities, or sponge storage facilities because of the effect water has in intensifying titanium fires.

274. Fire extinguishers approved for use on combustible metal fires, dry salt or other dry chemicals recommended for extinguishment or containment shall be stored in sufficient quantity in sealed drums or buckets in all areas where titanium fines and sponge are

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bored, crushed, dried, blended, or stored. Shovels shall be kept readily available adjacent to the stored salt, flux or powder. All extinguishment and storage areas shall be clearly identified with signs.

275. The use of inert gas (argon or helium) to extinguish titanium fires is only practical where the burning titanium is confined to a small space. This is not a practical method of extinguishment for large fires.

276. Segregation of titanium fines and sponge into noncombustible drums or tote-bin size increments is recommended for handling or storage. Burning drums or tote bins shall be moved away from processing equipment and out of buildings as rapidly as possible with fork trucks or other hauling equipment. As stated in Paragraph 274, application of dry salt or a dry powder will tend to minimize exposure to the fire and contain it, however, any container in which a fire occurs will usually become a total loss along with the material in it.

277. When a fire occurs in processing equipment further feed to the equipment shall be stopped; however, the equipment should be kept in operation, if possible, until all burning material is removed to reduce equipment damage. Although titanium produces intensive heat when burning, small amounts of burning material can be handled with a shovel to facilitate removal.

28. Personnel Safety Precautions.

281. Titanium tetrachloride in contact with moist air or water, hydrolizes to form hydrogen chloride gas, and hydrochloric acid. Hydrogen chloride fumes are toxic and highly irritating to the respiratory tract. If not removed immediately, titanium tetrachloride in contact with the eyes or skin will result in a double burn, one caused by the acid and the other caused by the heat of reaction.

a. All personnel exposed to titanium tetrachloride leaks or spills in titanium tetrachloride plants, storage and transfer areas, and laboratories should wear protective clothing, goggles or face shields and carry approved respiratory protection at all times. Respirators should be worn while drawing samples or making transfers.

b. A supply of clean soft rags should be kept available in clearly marked areas in titanium tetrachloride plants, analytical laboratories, and storage and transfer areas. Areas of skin coming into contact with titanium tetrachloride should be wiped immediately and then flushed with a large amount of water. Safety showers shall be installed at critical locations throughout the titanium tetrachloride plant, storage and transfer areas and laboratories.

c. Eyes splashed with titanium tetrachloride should be flushed immediately with large amounts of water. Eyewash fountains or eyewash bottles shall be located at critical areas.

d. Protection as outlined above also applies to the handling of caustic, hydrochloric acid, nitric acid, and sulphuric acid. Rubber coats and rubber gloves are recommended for handling acid.

282. Chlorine fumes are highly irritating to the eyes and respiratory tract. Personnel working in chlorine handling and storage areas should carry respiratory protection at all times. Canister-type gas masks are effective only to a maximum concentration of 1000 ppm. Liquid chlorine should be handled and stored in accordance with recommendations of the Chlorine Institute, Inc., as published in the Chlorine Manual.*

283. Personnel involved in reduction furnace tapping, removal of molten magnesium chloride, and magnesium refining operations should wear tight above-ankle shoes, fire-retardant clothing, heat-resistant gloves and face shields. Respirators should be carried at all times.

284. If contacted with water, molten magnesium presents a potentially dangerous fire and fume hazard, in addition to an explosion hazard. Tight above-ankle shoes, fire-retardant clothing, heat-resistant gloves and face shields should be worn by all personnel involved in magnesium refining and casting operations. Respirators should also be carried at all times. Magnesium refining and casting areas should be protected from rain, and all possibilities of water spillage avoided or eliminated.

285. Liquid sodium presents a potentially dangerous burn hazard. Personnel involved in the use, handling and storage of liquid sodium should wear protective shoes, clothing, gloves and face shields. Liquid sodium splashed on the skin should be wiped off with a soft cloth immediately and the affected area covered with mineral oil. Flooding with water to rapidly remove large quantities from the skin is recommended.

286. Magnesium chloride in the solid or dust state will readily absorb moisture to create a slipping hazard. Magnesium chloride dust on the skin can be highly irritating, especially if perspiration is

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present. Protective clothing, gloves, and respirators should be worn in high dust areas. Affected skin areas should be promptly washed with water.

287. Chemical safety goggles or face shields and protective clothing should be worn by all maintenance personnel when any joint or connection containing a potentially dangerous liquid or gas is being opened.

288. To prevent potential explosions caused by inadvertently using high pressure air in place of low pressure inert gas, fittings used on compressed air and inert gas line outlets should be different and not interconnectible.

289. It is suggested that the practice of wearing hard hats, safety shoes, and safety glasses be adopted and enforced at all times throughout a titanium sponge plant.

29. Sponge Storage and Shipping Regulations.

291. Long term storage of titanium sponge should be in 55-gallon steel galvanized drums with tight fitting clamp-on sealable lids. Short term storage may be in aluminum tote bins holding not more than 10,000 pounds.

292. Titanium fines should be stored in tightly sealed noncombustible 55-gallon drums.

293. Titanium storage areas shall be kept free of combustible materials, well ventilated, equipped with recommended fire protection equipment and plainly marked with "No Smoking" signs. Storage of drums shall be on steel pallets no more than two drums high.

294. The Office of Hazardous Materials, U.S. Department of Transportation, Washington, D.C., is currently charged with the responsibility of establishing shipping regulations on hazardous materials. At the present time there are no special regulations concerning the shipping of titanium sponge. However, it has been common practice to ship titanium sponge both within and without the United States in 55-gallon galvanized drums with tight sealed clamp-on lids.

295. Titanium powder and titanium fines are currently packaged and shipped according to specifications of the U.S. Department of Transportation.*

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CHAPTER 3. MELTING

31. Conversion of Sponge to Ingots.

311. Unlike many other metals that can be melted, cast or molded without unusual complications, titanium, because of its strong affinity for oxygen, hydrogen and nitrogen, and the tendency to become contaminated with other materials, must be melted in special water or NaK cooled copper crucibles under a medium high vacuum or under an inert gas blanket of argon or helium.

312. During the first stages of the development of the titanium industry, melting was accomplished with a nonconsumable electrode, usually carbon. The consumable electrode process using DC power was soon developed to meet quality and production requirements as commercial usage grew. However, nonconsumable copper electrode furnaces are now being utilized to reprocess scrap.

32. Melting Furnace Explosion Hazard.

321. During the 1950's several titanium melting furnace explosions occurred when water inadvertently entered the melting crucibles of furnaces during the melting operation. Three separate and distinct types of explosions are possible. An explosion may result from steam pressure, produced by water contact with molten metal, from a chemical reaction between molten titanium and water, or an explosion of free hydrogen, generated by a molten titanium-water reaction, may occur. If air is allowed to simultaneously enter the melting chamber as the result of a rupture due to a steam explosion, or some other type of failure, an air-hydrogen explosion of considerable force may result. All three types of explosions are considered possible in the same incident. This explosion hazard is present with both consumable and nonconsumable electrode furnaces using water as a cooling agent.

322. In 1955 representatives of five of the large producers of titanium and zirconium ingot formed an informal committee on safety practices in order to arrive at some basic recommendations for the benefit of the industry. The work of this committee has been published by the Defense Metals Information Center under the title, General Recommendations on Design Features for Titanium and Zirconium Production Melting Furnaces.*

a. Attention is called to the extreme importance of continuity of water supply for water-cooled crucibles. The water supply shall be continuously monitored by a system that will automatically

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interrupt power to the furnace at the instant of water flow decline and/or pressure drop. An emergency secondary source of water shall be provided adequate to cool the crucible in the event of failure of the primary source. The emergency system shall be cut in automatically by the same control that interrupts the power.

b. The use of a magnetic field to deflect the electric arc away from the crucible wall is recommended.

c. Water-cooled furnaces shall be located inside of protective concrete vaults or at least the crucible and water jacket portion shall be isolated to protect personnel and minimize damage in the event of explosion.

d. The furnace upper chamber shall be equipped with a relief device, such as special rupture disc, to aid in releasing pressure buildup in a safe manner if water enters the furnace. Means shall be provided to prevent reflux of air through the pressure relief port. The release pressure for the rupture disc should be set at approximately 20 psi above atmosphere. The use of low-pressure-release large explosion ports is not recommended.

e. Clearance of two inches or greater between the consumable electrode and crucible wall shall be maintained at all times by proper design and proper alignment of the electrode within the furnace and crucible.

f. The furnace shall be equipped with a device that continuously senses pressure within the furnace and will automatically interrupt power the instant the pressure rises to 5 psi or above.

g. The furnace shall be equipped with: 1) water flow sensors, temperature sensors and pressure indicators on all cooling systems; 2) arc voltage and amperage recorders; 3) electrode position and movement indicators; and 4) a furnace pressure sensor and recorder, with set point alarms on each system to warn of abnormal conditions.

323. Caution shall be used in handling and storing ingots that have been wetted during melting. Such ingots contain internal stresses that may cause them to shatter or literally explode, up to several days after the incident.

324. The use of the liquid metal NaK (sodium-potassium alloy) as a crucible coolant has been developed for both laboratory and commercial installations. The danger of furnace explosion from leakage into the melting zone is reduced. However, the handling of liquid metal has its inherent hazards. NaK reaction with water is violent. Recommended practices for shipping, handling and storing NaK may be obtained from MSA Research Corporation, Callery, Pennsylvania.

33. Casting.

331. The general process for shape casting of titanium is the "skull-casting" process wherein the material to be cast is melted as a consumable electrode in a tilting crucible. The power applied is normally somewhat higher than typical for ingot melting in order to develop a deep molten pool of metal. At the appropriate time in the melting cycle, the electrode is withdrawn and the casting poured. Vacuum or inert gas is provided for protection of the metal from atmosphere contamination.

332. The furnace crucible is made of copper with provision for water or liquid metal (NaK) cooling. Because of the high power levels used, seams in the crucible fabrication should not be visible to the electric arc or the molten metal. Suitable interlocks should prevent operation wtihout crucible coolant flow.

333. The molds for titanium casting may be of metal, graphite, ceramic, or a combination of these. In any event, the molds must be thoroughly dried, and stored carefully to prevent moisture accumulation.

334. Mold breaks are inevitable. Therefore, the furnace casting section must be cooled and/or sufficiently massive to accommodate a spill.

34. Fire Prevention.

341. Titanium sponge and alloys shall be stored in drums or tote bins with lids in place at all times.

342. Ordinary combustible material such as paper, wood, cartons, packing material, etc., shall not be stored or allowed to accumulate in sponge blending, melting, or casting buildings.

343. Residue from melting furnaces, especially from first melts of magnesium reduced sponge, shall be moved outside the building and placed in steel boxes for haulage to dumping areas. Collection boxes shall be kept a safe distance from all buildings. Other combustible waste materials shall be placed in a separate noncombustible pickup container and not mixed in with the melting residue.

344. Dry dust collectors used in collecting fines from blending, splitting and pressing operations shall be emptied at not less than daily intervals.

345. All sponge handling equipment shall be thoroughly cleaned, preferably by washing with water, before attempting any welding on the equipment or in close proximity. All welding should be controlled by a permit system.

346. All sponge handling and storage areas shall be clearly posted with "No Smoking" signs.

35. Fire Protection.

351. Extinguishers, such as water-base extinguishers, that are suitable for use on Class A fires are recommended for fires in ordinary combustible material. Extinguishers suitable for use on Class B fires are recommended for fires in oil, grease, and most flammable liquids. Extinguishers suitable for Class C fires should be used for fires in electrical equipment, and special extinguishing agents suitable for use on Class D fires should be used on metal fires. A water-base (soda-acid, foam, pressurized water) extinguisher should never be used on electrical or titanium fires. See NFPA Standards Nos. 10 and 10A.*

352. Small titanium fires may be controlled by the use of dry metal powder, flux or dry salt. (See Section 272.) Burning material should be removed from the equipment and the building as fast as possible. See Section 277.

353. Installation of automatic sprinkler systems in blending and melting buildings is not recommended.

36. Personnel Safety Precautions.

361. Water-cooled melting and casting furnace control consoles shall be remote from furnace melting areas, and outside of furnace vaults.

362. Personnel shall be prevented from entering vaults or furnace pits while water-cooled furnaces are melting.

363. It is suggested that personnel working around melting furnaces wear hard hats, safety shoes, and safety glasses at all times. Special fire-retardant clothing may be required when unloading or cleaning furnaces.

364. Personnel entering furnace shells to perform inspections or repair work should first make certain that any inert gas has been pumped or blown out and that all pyrophoric residue has been removed.

365. Personnel working around NaK cooled furnaces should wear face shields in addition to other protective clothing, when loading, unloading, or repairing furnaces.

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37. Ingot Storage and Shipment.

371. Titanium in ingot form presents no unusual hazard in shipment or storage. No special regulations exist concerning the shipment of ingots.

372. Since all commercial titanium ingots are round, special tongs are recommended for handling. When lying on the floor ingots should be kept in saddles to prevent rolling.

CHAPTER 4. MILL OPERATIONS

41. Forming and Shaping Processes.

411. Forging remains the most popular method of forming titanium because it is generally simpler and less costly than other forming processes. Gas or electric furnaces with accurate heat control are used to heat the metal into the proper forging range, which may vary from 1600° F to 2300° F. The rate of heat-up and final temperature must often be precisely controlled to achieve specific metallurgical and physical properties. Slabs, billet and bar stocks are produced by forging.

412. Large rounds of titanium are produced by lathe turning or grinding forgings. A considerable amount of titanium strip, coil and duct, down to foil thicknesses is produced from slabs on both continuous and hand mills. Wide sheets and plates of various thicknesses are produced on hand mills or plate rolling mills. Temperature control during rolling is important. Shearing and straightening operations are necessary to trim sheet and plate to size, also to straighten or flatten plate and straighten forged bar or extrusions. Titanium wire is produced from coils of rolled bar by drawing. Fastener stock is produced from coils of wire. Titanium tubing is produced by inert gas seam welding of rolled narrow strip. Heavy wall seamless tubing is extruded.

413. Special types of grinding operations are performed in mills. Swing grinders are used to spot grind ingots, slabs, billets and bar stock. Centerless grinders are used to finish round bar and fastener stock. Strip in coil form is ground continuously, and sheets are individually ground. Cold saws and abrasive cutoff saws are used to cut billet and bar stock to length. Swarf, or finely divided metal particles, are produced by all sawing and grinding operations.

42. Heat Treating.

421. Because of the potential for contamination, annealing of titanium without protection from air is performed in limited cases only.

422. Vacuum annealing is used to prevent contamination from the air and to remove hydrogen. Exhaust gases shall be vented to the outside atmosphere. Helium or argon should be used to backfill.

423. Many titanium alloys require heat treatment other than annealing such as solution treatment, quenching and aging. Quenching media may be cold metal block, inert gases, air, liquid salt baths, oil, water and aqueous solutions.

43. Pickling and Cleaning.

431. Scale formed on titanium during forging, hot rolling or other processing is now removed by pickling in sodium-potassium hydroxide, commercial descaling agents, or conventional acid pickling solutions such as hydrofluric, nitric and sulfuric acid. The fire hazard previously eminent when thin sheets were pickled in alkali salts has been eliminated.

432. A commercial water soluble detergent is commonly used for degreasing. This agent presents no fire or special personnel hazards.

433. Plate and extrusion bar is sometimes cleaned by grit blasting, creating a dust condition.

44. Fire Prevention.

441. Good housekeeping is required. Ordinary combustibles — packing materials, cartons, rags, pieces of wood and paper shall not be stored or discarded around mill equipment or in working areas of mill operations, especially dry grinding operations where hot sparks are prevalent.

442. Fuel lines to gas- or oil-fired furnaces or other heating equipment shall be equipped with emergency shutoff valves installed at an accessible location remote from the equipment being served. All lines and fittings shall be inspected regularly to detect corrosion or mechanical damage that might permit leaks to develop. See NFPA No. 86A.*

443. Open tanks on which flammable solvents are used for degreasing or pickling shall comply with NFPA No. 34.*

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444. When mobile bins on castors are used to transport oily crushed turnings, raw lathe turnings, or swarf, bin-to-floor ground straps shall be used to minimize possible sparking from static charge. Sawing, grinding, and cutting equipment should be well grounded. A static spark may easily ignite wet swarf. See NFPA No. 77.*

445. Oily crushed lathe turnings, raw turnings and swarf shall be collected in closed-top metal containers and removed daily to safe storage or disposal areas.

446. Forging presses, heavy grinders and other mill equipment operated by hydraulic systems shall use a nonflammable oil. However, use of this oil requires special seats in all valves and motors. Oil leaks shall be repaired and leakage kept to a minimum. Cleanliness is required in areas where hot metal is handled or sparks are produced and bits of scale continuously are being dropped. The danger of fire is great if a nonflammable fluid is not used.

447. No welding or torch cutting should be performed in the mill processing area unless authorized by a permit system. Smoking shall not be permitted in mill processing areas.

448. Nonflammable water soluble coolant shall be used for wet grinding, cutting and sawing operations. Coolant shall be filtered on a continuous basis and cake removed daily to a safe storage or disposal area. Finely divided titanium particles, particularly grinding swarf, are extremely flammable. Smoking shall not be permitted in operating, storage or disposal areas.

449. Dry grinding and cutting operations should be equipped with wet dust and fume collection equipment. Sludge shall be removed daily to a safe remote storage or disposal area. Fume ducts shall be flushed with water at regular intervals. See Sections 561 through 566.

45. Fire Protection.

451. Dry salt or dry powder approved for use on Class D fires is recommended for control of titanium fires.

452. Carbon dioxide extinguishers may be used to stop re-ignition of fires that often occur on small hot forgings or sheet during processing.

453. All mill buildings should be equipped with extinguishers suitable for use on electrical and refuse fires. However, all personnel should be instructed not to use this type extinguisher on titanium fires.

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46. Personnel Safety Precautions.

461. Personnel operating mill equipment should wear face shields, gloves and proper protective clothing wherever applicable.

462. The wearing of hard hats, safety glasses and safety shoes by all persons in the plant at all times is recommended.

463. In plants where it is common practice to burn swarf, great care should be exercised in the handling of the swarf, selection of the burning area and in controlling the method of ignition. This type of material has been known to explode shortly after ignition spreading fire to the transporting vehicle or mobile bin and a fairly large surrounding area.

47. Mill Product Storage and Shipping Regulations.

471. Finished products awaiting shipment should be stored in an orderly manner with adequate aisle spacing. The storage area should be kept free of combustible materials.

472. There are no special regulations covering the shipment of titanium mill products. Packaging, handling, and supporting of mill products under shipment should be in accordance with practices developed for other metals such as steel, copper, brass, and aluminum. Automatic sprinkler protection is recommended in warehouses packaging metal in paper casing and wood crates.

CHAPTER 5. MACHINING AND FABRICATION

51. Machining Operations.

511. Titanium may be turned, milled, bored, sawed, ground, abrasive cut with standard metal cutting or shaping equipment. Difficulties encountered during the early commercial development have now been overcome with the majority of fabricating and machining companies having learned to handle titanium in routine fashion.

512. Cutting tools must be of proper design and kept sharp for satisfactory work with titanium. The use of water soluble cutting oils has been considered standard for some years with no special problems of fires arising during machining operations. Improperly designed or dulled tools may produce high temperatures at the interface and cause ignition of turnings if a proper supply of coolant is not used.

52. Pressing and Forming.

521. A vast quantity of titanium parts are now produced by hot forging. Forging of large parts requires proper handling equipment and temperature controlled heating furnaces.

522. Titanium parts are also made by both hot and cold forming. No special hazards are involved in these fabrication techniques other than the normal equipment operating hazards.

53. Welding.

531. All welding of titanium metal must be performed under the protection of an inert gas, helium or argon, to avoid contamination from elements in the air. Welding of small assemblies is often carried out in inert gas filled chambers. Special shielding devices are required for larger assemblies where chambers are impractical.

54. Fire Prevention.

541. Small particles of titanium dust produced in dry sawing, grinding, machining or abrasive cutting are highly combustible and may present a fire or explosion hazard if allowed to accumulate where they can be accidentally ignited or dispersed to form a dust cloud. Work areas shall be cleaned daily and residue removed to a safe storage or disposal area.

542. Swarf accumulations from wet sawing, grinding, machining or abrasive cutting shall be cleaned up daily and removed to a safe storage or disposal area.

543. Combustible materials shall not be discarded in containers used for the collection of dust, swarf or turnings.

544. Combustible materials should not be stored in metal working areas. Oil spills should be cleaned immediately, particularly in the areas where dry grinding operations are taking place.

545. Smoking shall be prohibited in titanium metal working or scrap collection or storage areas.

546. No open flames or electric or gas cutting or welding equipment shall be used for repairing machinery or for other purposes in the shop area while the machines are in operation. If the use of cutting and welding equipment becomes absolutely necessary in making repairs, building additions, alterations, etc., all shop machines producing fines or dust shall be shut down and the entire section where the work is to be done shall be thoroughly cleaned by a water flush to remove all accumulations of fines, dust and other combustible material. This also applies to fume exhaust and collection system ductwork. All gas or electric cutting or welding operations shall be carried out under the observation of a responsible person who has adequate fire fighting apparatus at his disposal and is thoroughly trained in its use. He shall be assigned no other duties during the cutting or welding operation which he is to observe.

547. All scrap material shall be stored in accordance with the recommendations of Chapter 6.

55. Fire Protection.

551. Suitable extinguishment agents for titanium fires, such as those approved by Underwriters' Laboratories, Inc., for combustible metal fires shall be kept within easy reach of every operator performing machining, grinding or other operation on titanium. Suitable extinguishment powder should be kept in substantial containers with easily removable covers and a hand scoop provided at each container for applying the powder.

552. Containers of approved extinguishment powder or other extinguishment agents for titanium fires shall be plainly labeled.

553. Automatic sprinkler system protection is recommended in areas where titanium parts or assemblies are machined, fabricated, or stored. Finely divided titanium metal, or swarf, shall not be stored in the same area.

56. Dust Collection.

561. Dust should be collected by means of suitable hoods or enclosures at each dust-producing operation, such enclosures to be connected to a water precipitation type of separator, and the suction unit installed in such a way that the dust shall be converted to sludge without contact, in a dry state, with any high-speed moving parts. Figures 1 and 2 show typical water-crushed dust collectors servicing fixed and portable grinding units. Figure 3 shows diagrammatically four methods of dust precipitation used in the collectors shown in Figures 1 and 2.

562. Connecting ducts or suction tubes between dust collecting points and collection equipment shall contain water spray nozzles for washing out, and shall be completely bonded and grounded. Ducting and tubing shall be as short as possible, with no unnecessary bends. Ducts shall be carefully fabricated and assembled in accordance with NFPA No. 91.*

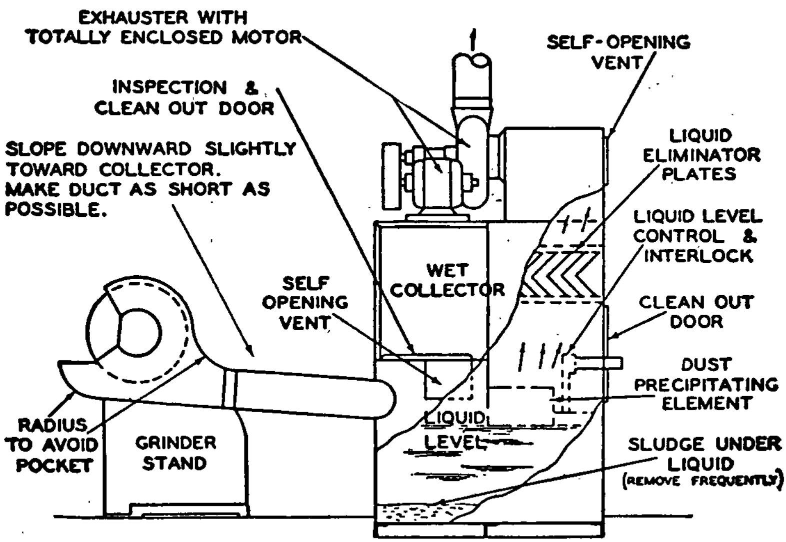

FIG. 1

NOTE: This drawing is schematic and intended only to indicate some of the features which should be incorporated in the design of a collector. The volume of all dust-laden air spaces should be as small as possible.

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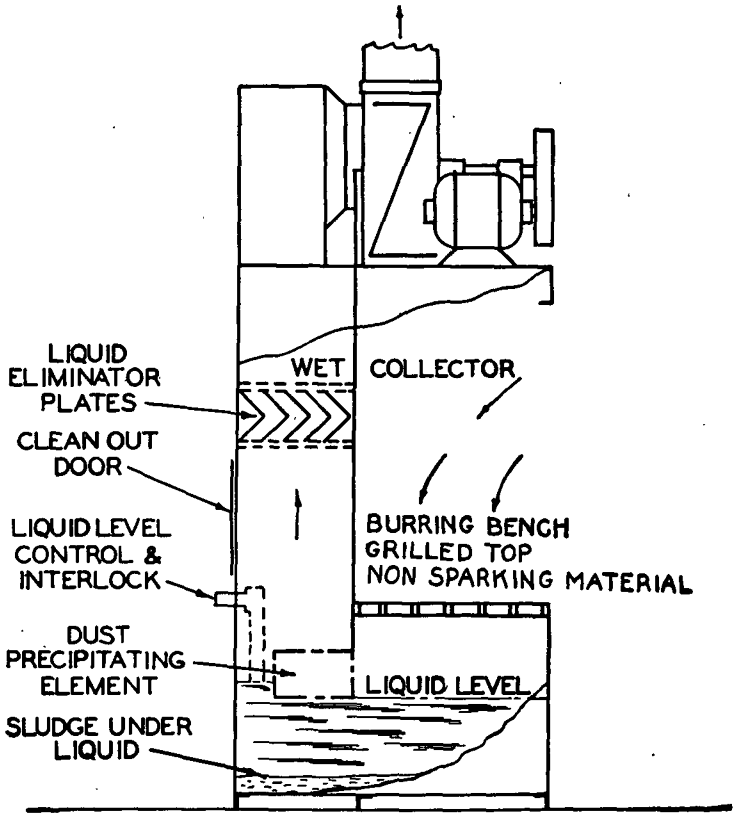

FIG. 2

563. Each source of dust shall be equipped with its individual dust-separating unit, except that with multiunit machines not more than two dust-producing units may be served by one separator. Not more than four portable dust-producing units in a single enclosure or stand may be served by one separator unit.

564. The power supply to dust-producing equipment shall be interlocked with the motor driving the exhaust blower and the liquid level controller of the wet collector in such a way that improper functioning of the dust collecting system will shut down the equipment it serves. A time delay switch or equivalent devices

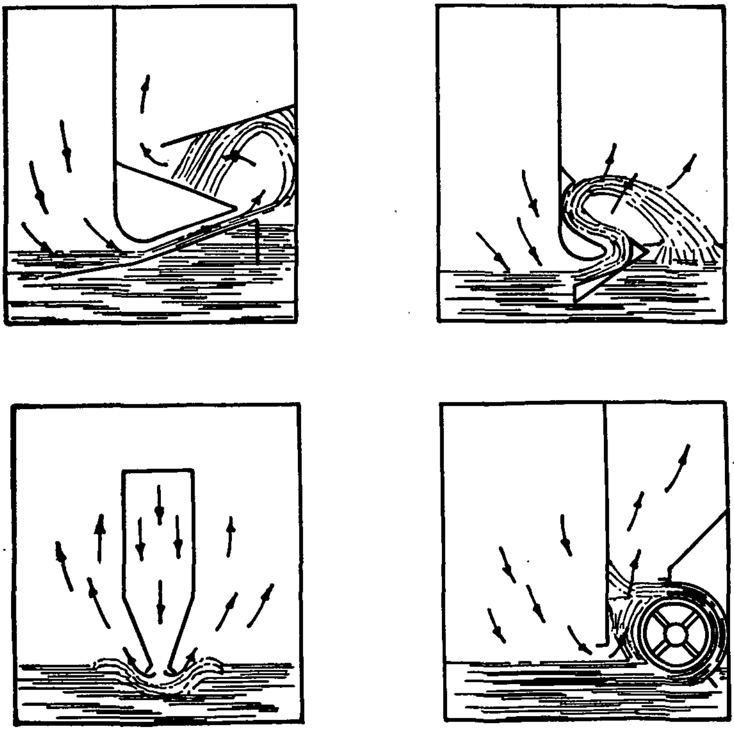

FIG. 3

shall be provided on the dust producing equipment to prevent the starting of its motor drive until the wet collector is in complete operation and several changes of air have swept out any residual hydrogen.

565. Systematic cleaning of the entire building area containing dust producing equipment, including roof members, pipes, conduits, etc., shall be conducted daily or as often as conditions warrant, (1) by use of soft brushes and nonsparking scoops and containers, or (2) by means of a fixed suction pipe and outlet vacuum cleaning system, provided the separator unit is of the water-precipitation type and provided also that the suction piping system consists of standard mild steel pipe and standard recessed drainage fittings, with a check valve installed at each outlet. Implements and hose used in connection with stationary vacuum systems shall be bonded and grounded. See NFPA No. 77.* A rupture diaphragm shall be provided in the piping at its connection to the inlet side of the separator in such a way that a possible explosion in piping may be safely vented to the atmosphere.

566. Sludge from dust separators and vacuum cleaning unit precipitators shall be removed daily. Covered, vented steel containers shall be used to transport the collected sludge to a safe point for storage, or for disposal by mixing with sand and burying or burning by locally approved methods.

NOTE: Sludge shall be burned in a segregated area at a safe distance from combustible material. The burning area may be a layer of fire brick or hard-burned paving brick with sufficient slope to permit it to drain properly. The wet sludge should be spread in a layer not over three or four inches thick. Ordinary burnable refuse is placed over the wet grinding sludge and ignited. The burning refuse supplies enough heat to dry and ignite the top surface of the sludge and the entire pile burns.

57. Personnel Safety Precautions.

571. All machines shall be properly equipped with required safety guards.

572. The wearing of safety glasses and protective clothing shall be mandatory for all machine operators. Maintenance personnel and material handlers should also be required to wear hard hats and safety shoes.

573. All working area and aisleways shall be properly lighted and kept free of stored materials.

574. Personnel working in dusty areas or operating equipment creating fine particles of metal or dust shall wear respirator protection.

575. Welding and cutting areas shall be properly ventilated.

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