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D. L. COURSEN ETAL

3,022,544

EXPLOSIVE COMPACTION OF POWDERS

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2 Sheets-Sheet 1

FIG. 1

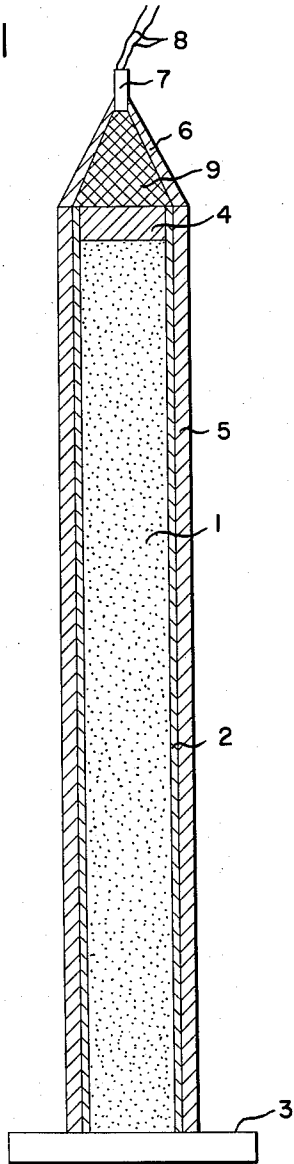
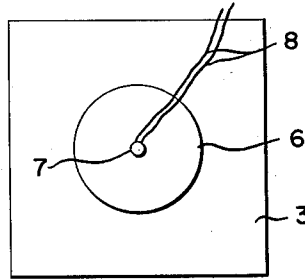


FIG. 2



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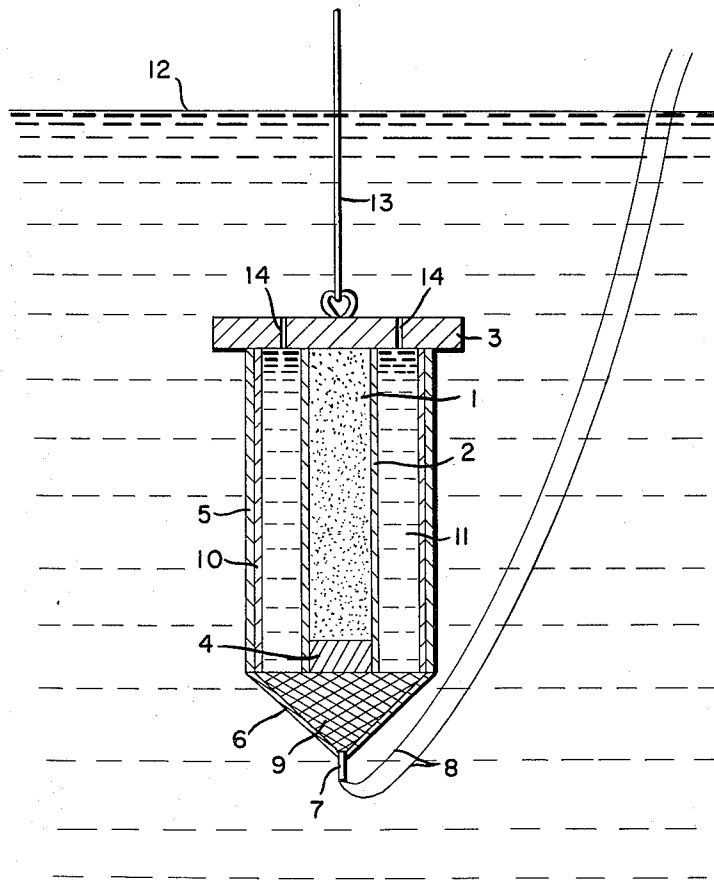
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FIG. 3



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EXPLOSIVE COMPACTION OF POWDERS

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2 Claims. (Cl. 18—59.3)

The present invention relates to the forming of compacts from powdered material. More particularly, this invention relates to a novel method for compacting powders whereby compacts of unusually high density and strength are obtained and whereby large compacts can be produced.

This application is a continuation-in-part of our co-pending application serial No. 697,614 filed November 20, 1957, now abandoned.

The techniques of powder metallurgy are widely employed in the processing of metals melting at high temperature, sometimes referred to as refractory metals, and in the preparation of combinations of metals that cannot otherwise be obtained because the metals are not miscible or because of very wide differences in melting point. In essence, the technique involves compression of the loose metal powder into a consolidated mass which can be further processed. In most cases, such further processes involve first a sintering operation to make the mass coherent. In the case of many materials, the coherent mass thus prepared can be rolled or drawn directly to the desired product. In other cases, the sintered compact must be further processed before the metal can be fabricated into finished products. In either case, the quality of the compact is largely dependent upon the uniformity and degree of compression obtained in the first step. The strength of the sintered compact is directly dependent upon the density of the compact.

At present, most compacts of powdered metals are produced by introducing the loose powder into a die and compressing it by a mechanical press. This operation poses many problems. For example, the compressed metal will have a density of from 3 to 15 times that of the loose powder; thus the stroke of the press and the size of the die must be sufficient to compensate for the reduction in volume occurring during compaction. This factor alone imposes a severe limitation on the size of the compact which can be prepared, since presses having very long strokes are not feasible, particularly when very high pressures, i.e., up to 200 tons per square inch, are required. The compacting load in a press is proportional to the area of the powder being compacted. Therefore, for an average compacting pressure of only 50 tons per square inch, a 500 ton press could press a compact of only 10 square inches. Obviously, to press compacts of large surface area, enormous presses are required. When pressures of 200 tons per square inch are required and large surface areas are involved, the mechanical press becomes unfeasible. Removal of the compact formed from the die represents a further problem with some metals since the compact has a tendency to expand when removed from the die. Thus, as the compact is being removed, that portion of the compact outside of the die expands while the portion within the die cannot; this action causes cracks in the compact. Provision must be made for the escape of air from the die during the compression without permitting the powder to be blown out. The die walls must be smooth to facilitate removal of the compact and sufficiently heavy to withstand the tremendous pressures involved. In addition, many powder compositions contain hard and abrasive particles. When such compositions are mechanically pressed, the life of the die is very short and frequent replacement is required. Thus,

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many mechanical problems exist and expensive equipment is required for mechanical pressing of metal powders.

Another factor of great importance lies in the fact that the resistance of the powder to compression increases rapidly with the depth of the powder column. Because of inter-particle friction as well as friction with the die walls, a considerable variation of density will occur if an attempt is made to form a compact more than a few inches in depth. In a number of applications, this limitation presents a very serious handicap. This is particularly true in the case of the refractory metals which must be further processed by a melting operation.

As previously mentioned, the compressed powder must be sintered in order to provide sufficient strength for ordinary handling. This sintering may be performed by external heating or by internal heating of the compact caused by passage of an electric current through the compact. The temperatures involved are slightly lower than the melting temperature of any of the components of the compact so that the powder particles do not fuse or melt, but the solidity of the compact results from the rearrangement of individual particles and their growth at the expense of neighboring particles. Furthermore, it has been found to be both difficult and costly to fabricate certain metals such as titanium and niobium by conventional methods.

The present practice is to produce electrodes by hand-welding a number of compacts together and then forming an ingot by an arc-melting process in which the electrode is consumed. Since pressed compacts are of irregular density, the ingot produced usually contains flaws, and the process is repeated using the first ingot as the electrode in order to provide a billet or slab suitable for fabricating finished products. Also, the density of the compact directly affects the quantity of metal in the electrode—if the density is low, a large electrode will produce a small ingot and several ingots may have to be welded together to produce a billet or slab of the desired size. Experience has shown that continuous arc-melting is required to produce a satisfactory billet, therefore a billet of desired size cannot be prepared by melting two or more unjoined electrodes one after the other.

The described techniques of powder metallurgy are applied to many materials other than pure metals. The preparation of compacts of metal compounds such as metal oxides, metal carbides, metal nitrides, and metal borides is widely practiced in view of the growing needs for articles prepared from such materials. Nonmetal powders such as carbon, silicon, organic compositions, and polymeric materials are also compacted by the described techniques. Mixtures of metals and other materials such as binders, lubricants, etc., are compacted to provide compositions having special properties. The so-called self-lubricating bearing is an example of this type of compact. To a greater or lesser degree, the problems of pressing these materials are as severe as are those for the metal powders.

From the foregoing, the inadequacy of the present procedures is apparent. The preparation of small compacts by pressing and their joining by hand welding is very time-consuming and uneconomical. The low-density compacts obtained by the conventional pressing methods require excessive further processing, thus adding still more to the time and expense required to convert the metal powder into a fully usable form. Thus the need for a better method of preparing metal compacts is obvious.

In U.S. Pat. 2,648,125, a method of preparing compacts is described which involves loading the powder into an impervious bag, immersing the filled bag in water contained within a heavy walled vessel, and then compacting the powder by the hydrostatic pressure produced

by the action of a piston on the body of water, the piston being driven by the gases produced from a deflagrating explosive charge. According to the patent description, uniform pressures of about 50,000 pounds per square inch can be obtained and a uniform compact is thus produced. In an article published in *Business Week* (September 20, 1952), the preparation of compacts by immersing a bag filled with powder in water contained within a 14-inch cannon breech block and detonating a dynamite charge within the sealed breech block is also described. To some degree, the foregoing methods overcome the disadvantage of mechanical presses by eliminating a die and providing a uniform pressure on all sides of the powder being compacted. However, the size limitation remains because it is not practicable to build a very large vessel capable of withstanding pressures of 50,000 pounds per square inch, and the maximum pressure which can be applied to the compact is limited because of the requirement of a vessel capable of withstanding the pressure produced.

Accordingly, an object of the present invention is to provide a method for compacting powders wherein all of the above described disadvantages are overcome. A further object is to provide a method for preparing compacts of essentially theoretical density. A still further object is to provide a method for preparing compacts whereby unique results can be obtained. Additional objects will become apparent as this invention is more fully described.

We have found that the foregoing objects are achieved when we prepare a compact by surrounding a mass of the powder with a layer of a high-velocity detonating explosive and then detonate the explosive charge. In order to obtain uniform compaction of the powder, the layer must have a substantially uniform quantity of detonating explosive per unit of area. Furthermore, if the explosive layer is initiated at one point only, shock waves converging at the surface 180° from the point of initiation will be produced. The higher pressures at the converging front introduce stresses in the compact which may cause cracking. Accordingly, therefore, we prefer to initiate the explosive layer at a sufficient number of locations to avoid the production of converging shock waves. The most satisfactory initiation is provided when the entire periphery at one end of the layer is simultaneously initiated.

For ease of handling and to prevent loss of material, the powder to be compacted is preferably encased in a container. Inasmuch as a seam of a container represents a discontinuity in its surface, such seam will introduce a defect on this surface of the metal compact; thus, we prefer to use a seamless container. By using a container of sufficiently heavy walls, wrinkling of the container and consequent surface irregularities of the compact can be avoided—the container will be uniformly compressed to compensate for the reduction in volume accompanying the compaction of the powder. In order to eliminate the need for removing the container after compaction, a container fabricated from the same composition as the powder being compacted may be used.

In order to illustrate the carrying out of the method of the present invention, reference is now made to the accompanying drawings in which FIGURE 1 is a sectional lengthwise view of an assembly for compacting powder, and FIGURE 2 is an end view of the same assembly and FIGURE 3 is a side view partially in cross section illustrating further embodiments of the method of the present invention. In FIGURE 1, 1 represents a mass of powder within tube 2 fastened at one end to base 3 and closed at the other end with stopper 4. 5 represents a wrap of a high velocity detonating explosive around tube 2, 6 is a layer of a detonating explosive around inert cone 9, and 7 is a conventional blasting cap having electrical lead wires 8.

In carrying out the method of the present invention,

when cap 7 is actuated, explosive layer 6 is initiated. Because of the conical configuration of layer 6, explosive wrap 5 is initiated simultaneously all along its edge adjacent to layer 6, and the detonation proceeds along the length of wrap 5 without the formation of any undesirable converging shock waves. The detonation of wrap 5 constricts tube 2 and compresses powder 1.

FIGURE 3 illustrates the interposition of a water annulus between the powder to be compacted and the layer of high explosive and also the submergence of the compaction assembly in an unconfined body of a dense fluid, for example water. In this figure, elements 1 to 9 are as in FIGURES 1 and 2. In accordance with one embodiment of the invention illustrated in FIGURE 3, explosive layer 5 surrounds a retainer element, e.g. a can, 10, which maintains the water annulus 11 between the mass of powder 1 and explosive layer 5, retainer 10 being fastened, e.g. by welding, to base plate 3. Conical stopper 9, which is surrounded by explosive layer 6, is fastened to retainer 10. In accordance with a further embodiment of the invention, the assembly is shown submerged in an unconfined body of water, the surface of which is indicated by 12. The assembly is suspended in the water by suspending-retaining means 13, shown as a chain attached to an eye hook fastened to base plate 3. Optionally, channels 14 may be provided, as shown, in base 3 when the assembly of the water-annulus technique is to be submerged in water, to permit filling of the space between powder container 2 and water-retainer 10 with water.

In order to illustrate further the present invention, reference is now made to the following examples. In the examples, unless otherwise indicated, the explosive wrap consisted of sheet explosive prepared by blending 85 parts of PETN, 7.5 parts of butyl rubber, and 7.5 parts of a thermoplastic terpene resin (mixture of polymers of β -pinene having the formula $(C_{10}H_{16})_n$, commercially available as "Piccolyte" S-10 manufactured by the Pennsylvania Industrial Chemical Corporation), and rolling the blend into sheets, the thickness of the sheet determining the weight per unit of area. The composition has a velocity of detonation of about 7200 meters per second and the sheets are strong, flexible, and nonresilient.

Example 1

A seamless tube of cold-drawn steel having a length of 8 inches, an inner diameter of 1½ inches, and a wall thickness of ¼ inch mounted on a steel base plate 3 inch x 3 inch x ½ inch was filled to within ½ inch of the top with titanium sponge, the tube being vibrated during loading. The titanium sponge thus packed had a bulk density of about 1.5 grams per cubic centimeter. A rubber stopper ¾ inch in length was inserted in the open end, and a clay cone 1¼ inch in length was placed on the stopper. The tube was wrapped with the described explosive sheet, the explosive load being 2 grams per square inch. A sheet of the same composition but containing 4 grams of explosive per square inch was used to form the layer about the cone, and this layer was joined to the wrapping around the tube by taping. A commercial detonator (a number 8 electric blasting cap) was fastened to the apex of the conical layer, and the assembly was immersed in water. The blasting cap was initiated by application of an electric current, causing detonation of both layers of explosive. The assembly was recovered and the constricted outer tube was removed.

The compact produced was very strong, had the appearance of a solid metal rod, a diameter of 1 inch, and a density of 4.36 grams per cubic centimeter, 97% of the theoretical density.

Example 2

The procedure of Example 1 was repeated using dendritic titanium powder in place of the titanium sponge.

The compact had a diameter of $1\frac{1}{16}$ inch and a density of 4.40 grams per cubic centimeter, 98% of the theoretical density. The compact was strong and had the appearance of a solid metal rod.

Example 3

The procedure of Example 1 was followed by using atomized aluminum powder (60% through a 325-mesh screen) packed in a stainless steel seamless tube 6 inches in length, $2\frac{1}{4}$ inches in inner diameter, and $\frac{1}{16}$ inch in wall thickness. The packed powder had a bulk density of about 1.5 grams per cubic centimeter. The compact produced had a density of 2.65, 98% of theoretical density, and was completely free of cracks. The diameter of the compact was $1\frac{1}{16}$ inches. A transverse slab cut from the compact was used to determine tensile strength and gave a measurement of 5,400 pounds per square inch.

Example 4

The procedure described in Example 1 was used to compact copper powder having a bulk density of 4.1 grams per cubic centimeter in a cast iron tube $1\frac{1}{16}$ inches in inner diameter, 7 inches in length, and $\frac{1}{8}$ inch in wall thickness by using an explosive load of 3 grams per square inch. The compact produced had a density 97% of theoretical (8.92 grams per cubic centimeter) and a diameter of $1\frac{1}{16}$ inch. The center was unconsolidated, the diameter of the unconsolidated portion being about 0.05 inch.

Example 5

The procedure described in Example 1 was used to compact electrolytic iron powder, which had been vibrator-packed into the compaction tube to a bulk density of about 3.8 grams per cubic centimeter. The results are as indicated in the following table.

Type	Tube			Explosive loading (g./in.)	Compact		
	Length (in.)	I.D. (in.)	Wall (in.)		Diameter (in.)	Density (g./cc.)	Percentage of theoretical density
CDS	23	$1\frac{1}{8}$	$\frac{1}{16}$	4	$1\frac{1}{8}$	7.7	98
SS	8	$2\frac{1}{4}$	$\frac{1}{16}$	6	$1\frac{1}{8}$	7.7	98
SS	8	$3\frac{3}{16}$	$\frac{3}{32}$	8	$2\frac{1}{16}$	7.7	98
SS	10	6	$\frac{1}{4}$	11	$4\frac{1}{4}$	7.6	97
CDS	12	8	$\frac{1}{4}$	16	$5\frac{1}{2}$	7.7	98
EA	6	$2\frac{3}{4}$	$\frac{1}{8}$	8	$1\frac{1}{8}$	7.7	98
Nylon	6	$1\frac{1}{2}$	$\frac{1}{8}$	2	$2\frac{3}{32}$	7.6	97

CDS—cold-drawn; SS—stainless steel; EA—extruded aluminum.

Example 6

The procedure of Example 1 was followed to compact niobium powder (through 30-mesh, held on 80-mesh screen) in a $37\frac{1}{2}$ inch long tube of cold-drawn steel having an inner diameter of $\frac{1}{4}$ inches and a wall thickness of $\frac{1}{16}$ inch, the explosive loading being 5 grams per square inch. The diameter of the compact was 1 inch, and the density was greater than 95% of theoretical. The compact was sufficiently strong to be used directly as a consumable electrode for the further processing of the niobium.

This particular powder could not be consolidated by mechanical presses applying up to 30 tons per square inch. Higher mechanical pressures were not feasible because of the excessive wear on the dies due to the hardness and abrasiveness of the niobium particles.

Example 7

The procedure of Example 1 was followed to compact crystalline uranium oxide (UO_2) packed to a density of 7.1 grains per cubic centimeter in a cold-drawn steel tube 8 inches in length, $1\frac{1}{4}$ inches in inner diameter, and $\frac{1}{16}$

inch in wall thickness. The results obtained were as follows:

	Compact explosive loading (g./sq. in.)	Compact diameter (inches)	Density (g./cc.) ¹	
			Peripheral	Center
A	2	$1\frac{1}{8}$	8.8	7.6
B	4	$1\frac{3}{16}$	9.6	8.1
C	8	$1\frac{1}{2}$	10.0	9.6

¹ The theoretical density of uranium oxide is 10.9 grams per cubic centimeter.

Compact A had the brown color of the uncompact powder, approximately a $\frac{3}{4}$ inch diameter central portion was unconsolidated; compact B was also brown and had a center about $\frac{3}{8}$ inch in diameter of unconsolidated powder; compact C had a black, metallic color and no unconsolidated portion.

Example 8

This example illustrates the preparation of a beryllium compact following the procedure of Example 1. Beryllium powder, particle size about 50 microns, when vibrator packed to a density of about 0.6 gram per cubic centimeter in a seamless steel tube 8 inches in length, $2\frac{1}{8}$ inches in diameter, and $\frac{1}{16}$ inch in wall thickness, and compressed by the detonation of an explosive loading of $\frac{1}{2}$ gram per square inch, undergoes a reduction to approximately $\frac{1}{3}$ its original volume, i.e., to a diameter of $1\frac{21}{32}$ inch, resulting in a coherent compact having a density approximately 95% of theoretical.

Example 9

Tungsten powder was vibrator packed to a density of 7.3 grams per cubic centimeter in a cold-drawn seamless steel tube $1\frac{1}{4}$ inches in inner diameter, $\frac{1}{16}$ inch in wall thickness, and 8 inches in length. A sheet of explosive having a loading of 8 grams per square inch was wrapped around the tube, and the procedure described in Example 1 was followed to compact the powder. The diameter of the compact obtained was $4\frac{9}{16}$ inch and the compact was at 97.82% of theoretical density (19.3 grams per cubic centimeter). The compact had sufficient strength to withstand ordinary handling.

Example 10

Substituting zirconium powder (bulk density—2.8 grams per cc.) for the tungsten powder in the compaction described in Example 9 permits reduction of the explosive loading to $2\frac{1}{2}$ grams per square inch to achieve a compact having a density 95% of theoretical (6.4 grams per cubic centimeter) at a diameter of $1\frac{3}{16}$ inch.

Example 11

Silicon, obtained in the form of needles, was ball-milled and vibrator packed into a $2\frac{7}{8}$ inch inner diameter steel tube 8 inches in length and $\frac{1}{16}$ inch in wall thickness. Following the procedure of Example 1, using an explosive loading of $2\frac{1}{2}$ grams per square inch, a compact having a diameter of $2\frac{3}{16}$ inches was obtained. The density was 95% of theoretical (2.4 grams per cubic centimeter).

Example 12

Dendritic chromium powder was compacted to 95% of theoretical density (7.1 grams per cubic centimeter) by the procedure described in Example 1. The chromium powder, vibrator packed to a density of 1.8 grams per cubic centimeter in a seamless steel tube having an inner diameter of $1\frac{1}{8}$ inches, a wall thickness of $\frac{1}{16}$ inch and a length of 8 inches was compacted using an explosive loading of 3 grams per square inch to a diameter of $1\frac{1}{16}$ inch.

Example 13

A stainless steel tube 8 inches in length, $2\frac{1}{4}$ inches in inner diameter, and $\frac{1}{16}$ inch in wall thickness was packed

with $3\frac{1}{2}$ inches of copper powder and $3\frac{1}{2}$ inches of atomized aluminum powder. The procedure described in Example 1, an explosive loading 2 grams per square inch being used, was followed to form the compact. The compacted aluminum and copper were at 98% of their theoretical density, and the two metals were firmly joined to each other.

Example 14

A powder mixture consisting of 60 parts by weight of electrolytic iron, 30 parts of atomized aluminum, and 10 parts of copper was compacted by the procedure of Example 1 in a stainless steel tube $2\frac{1}{4}$ inches in inner diameter, $\frac{1}{16}$ inch in wall thickness, and 8 inches in length by using an explosive loading of 4 grams per square inch. The compact had a diameter of $1\frac{5}{16}$ inches.

Example 15

The procedure used in Example 1 was followed to prepare a compact of a steel alloy powder commercially sold for use in powder fabrication. The alloy has a composition of 0.5% carbon, 0.35% silicon, 0.75% manganese, 0.010% sulphur, 0.01% phosphorus, 1.85% nickel, 0.25% molybdenum, and the balance iron. In normal powder metallurgy practice, this powder is compacted at 30-50 tons per square inch and sintered at 2000 to 2100° F. To obtain greater strength and ductility, the sintered parts are reprocessed by sizing and resintering. This procedure should result in a compact having approximately 95% of theoretical density.

When compacted by an explosive loading of 5 grams per square inch in a $1\frac{5}{8}$ inch inner diameter, 6-inch-long cold-drawn steel seamless tubing having a $\frac{1}{16}$ inch wall thickness, the compact produced had a diameter of $1\frac{3}{16}$ inch and a density 97% of theoretical without sintering.

Example 16

A compact consisting of an iron core surrounded by an aluminum sheath was prepared by centering a thin-walled tube of $\frac{1}{2}$ -inch diameter with a cold-drawn steel seamless tube $1\frac{5}{8}$ inches in inner diameter ($\frac{1}{16}$ inch in wall thickness), filling the thin-walled tube with electrolytic iron powder and the annular space around the thin-walled tube with aluminum powder the tubes being vibrated during the filling operation. The thin-walled tube was then removed, and the assembly compacted, by using explosive sheet at a loading of 2 grams per square inch. The compact was free of cracks, had a diameter of $1\frac{1}{4}$ inch, and a very strong bond existed between the two metals at their interface.

Example 17

The procedure of Example 1 was followed using a powdered niobium alloy composition consisting of 75% niobium, 15% molybdenum, and 10% titanium. Satisfactory compacting was obtained.

The foregoing examples illustrate the application of the method of this invention to a variety of powders. In essence, the present method can advantageously be used in the compacting of any powder that can be mechanically compacted, and to powders that cannot be mechanically compacted. Because the explosive sheath can be made as long as desired, no limit exists with respect to the length of the compact which can be thus produced. As shown by the examples, excellent iron compacts of $5\frac{1}{2}$ inches in diameter were prepared. There is no reason to believe that such diameter represents nearly the maximum diameter compact which can be produced, particularly since an explosive loading of only 16 grams per square inch was required. The examples further show that the compacts obtained are of much higher density than that obtainable by conventional procedures, even including sintering. Inasmuch as the strength and working characteristics of a compact are directly related to its density, the obtaining of such high densities in a single operation is of tremendous significance. For many fabrications,

the compact thus formed can be used without further processing. When further processing is desired, such as for example in preparing billets from the compact by the consumable electrode process, the quantity of material in an electrode of a specified size is greatly increased by the higher density, and the higher strength and uniform distribution of the material causes a more regular and uniform melting zone. This latter feature may permit elimination of a second remelting operation in order to obtain a uniform billet.

The very high densities, usually almost theoretical, of the compacts are believed due to the tremendous pressures produced by the detonation wave itself acting on the powder. Calculations have indicated that the pressure of a detonation wave at the surface of a high-velocity detonating explosive charge are somewhere near 200,000 atmospheres. As the detonation wave progresses outwardly from the explosive charge, it normally is rapidly attenuated and the pressure drops rapidly. For example, if a spherical charge is centrally initiated, the shock wave produced issues as a spherical wave. The area of the sphere increases as the square of the radius, and therefore, the pressure must correspondingly decrease. However, when the explosive charge surrounds the object, the pressures directed toward the center are reinforced rather than attenuated. Thus, while the compaction of the metal absorbs energy rapidly, the concentration of the shock wave towards the center provides the energy needed to compact beyond the surface.

That the radially symmetric application of the pressure of the detonation wave is responsible for the high density obtained is fully illustrated by the following comparative examples.

Example 18

Two aluminum electric blasting cap shells (0.260 inch inner diameter, 0.010 inch wall thickness) were vibrator packed with electrolytic iron powder and the end of the shell sealed by crimping in a rubber plug. One shell was wrapped with explosive sheet in accordance with the present invention, the total explosive load (PETN) being equal to 1.6 grams. The assembly was submerged in an open body of water and the explosive sheet initiated by means of a No. 6 commercial electric blasting cap. The compact produced had a density of 7.68 grams per cubic centimeter, 97.5% of theoretical.

The second shell was placed in a bomb, a quantity of the same sheet equal to 1.6 grams of explosive and a No. 6 electric blasting cap were placed together in the bomb, and the bomb was filled with water and sealed shut by a rubber-gasketed cap plate bolted in place, provision having been made for the passage of the lead wires to the outside of the bomb. The cap was initiated, in turn detonating the PETN sheet. The compact thus formed had a density of only 6.23 grams per cubic centimeter, i.e., 79.5% of theoretical density.

The foregoing indicates clearly that the unfocused hydrostatic pressure produced by the detonation of an explosive in a confined body of water is much less than the pressure exerted by the same amount of explosive when the explosive surrounds the material being compacted. In the foregoing experiments, only very small compacts were prepared because of the requirement of a bomb having sufficient strength to withstand the pressures produced by even the small quantity of explosive used. The hydrostatic pressures on the walls of the bomb (as well as on the walls of the shell containing the compact) were about 50,000 pounds per square inch.

The beneficial effect of focusing the pressure produced by the detonation of the explosive charge does not depend upon the explosive being in contact with or even adjacent to the material being compacted. The following example illustrates the foregoing.

Example 19

Tungsten powder was vibrator packed to a density of

about 7.3 grams per cubic centimeter in a cold-drawn seamless steel tube 1¼ inches in inner diameter, ¼ inch in wall thickness, and 8 inches in length. The bottom of the tube was welded to a ¼ inch thick steel disk 4 inches in diameter, and the top was sealed with a rubber stopper. The assembly was then placed in a can having a diameter of 4 inches and a wall thickness of ¼ inch. The annular space between the can and the tube was filled with water, and the end of the can was closed by a conical stopper. Sheet explosive having an explosive content of 4 grams per square inch was wrapped around the can and cone, the assembly was submerged in water and the explosive was initiated at the apex of the cone by a No. 8 commercial blasting cap.

The compact produced had a diameter of 2½ inches and 98% of theoretical density. The compact was free of cracks and of sufficient strength for rough handling.

However, if desired, the explosive may be in contact with the powder being compacted. The following example illustrates such procedure.

Example 20

A sheet of explosive having an explosive loading of 2 grams per square inch was fitted inside a steel tube 1½ inch in inner diameter, ¼ inch in wall thickness and 8 inches in length so that the inner surface of the tube was covered at all points by the explosive sheet. Iron powder was then vibrator packed into the explosive-lined tube to a bulk density of about 3.8 grams per cc. The top of the tube was sealed with a conical rubber plug and a layer of 4 grams per square inch explosive sheet wrapped around the cone and about ½ inch of the tube adjacent to the cone. The assembly was submerged in water and the conical explosive layer initiated with a No. 8 electric blasting cap. The detonation of the layer overlapping the tube initiated the explosive within the tube. The tube itself was shattered, but a compact of iron powder having a diameter of 1 inch and a density 95% of theoretical was recovered. The compact showed a surface irregularity corresponding to the line formed by the meeting edges of the sheet explosive—such irregularity could be eliminated by using extruded explosive tube instead of sheet explosive.

In order to provide the high pressures required for compaction in accordance with the present invention, the explosive must detonate at high velocity. An unconfined deflagrating explosive could not provide the desired pressures. By the term "high-velocity detonating explosive," we mean a composition having, when unconfined, a velocity of detonation of at least 1200 meters per second. The selection of the particular high-velocity detonating explosive to be used will be dependent upon the size of the compact to be prepared. Obviously, the quantity of explosive in the layer surrounding the compact must be sufficient so that the detonation will be propagated throughout the entire length of the layer. In the case of small-diameter compacts, the quantity of explosive per unit of area required is so small that the less sensitive explosives such as RDX, HMX, TNT and the like would not propagate the detonation. On the other hand, the more sensitive explosives, such as PETN and nitroglycerin-based explosives, which will propagate detonation even in a thin layer will also propagate detonation in thick layers. Thus, when large-diameter compacts are desired, a much wider range of explosive compositions can be used.

In the preceding examples, a PETN-binder composition formed as a flexible sheet was used to provide the compacting energy. This form of explosive represents a preferred form because of the ease of application and control of loading density. The following example illustrates the use of blasing gelatin (91% nitroglycerin, 8% nitrocellulose, 1% chalk).

Example 21

A cold-drawn steel seamless tube 1½ inch in inner

diameter, 8 inches in length, and ¼ inch in wall thickness mounted on a 3 x 3 x ½ inch base plate was vibrator-packed with electrolytic iron powder, and the open end was sealed with a rubber stopper. A clay cone was taped to the tube, and the surface of the tube and clay cone coated with a ½-inch-thick layer of blasing gelatin (about 7 grams per square inch). The blasing gelatin was initiated at the cone apex by means of a No. 6 electric blasting cap. The compact produced had a diameter of 1¾ inch and was at 97.7% of theoretical density. This explosive loading represents a considerable overloading and the effect of the overloading was to produce a fused area in the center of the compact due to the concentration of the shock waves, this fused area resulting in a small axial channel.

As previously mentioned, a compact having a good appearance can be obtained only by initiating the explosive layer in such a way that the shock waves from the detonation converge only at the center of the compact. If only one point on the periphery of the explosive layer is initiated, the detonation will be propagated around the periphery as well as axially, and the detonation fronts will converge in a line 180° from the point of initiation. This convergence of detonation waves will produce pressures along this line much greater than those elsewhere on the surface of the compact causing distortion, if not cracking. Further, the density within the compact will not be uniform in the absence of the centrally converging shock wave. As shown by the procedure described, the formation of the undesired converging shock wave can be avoided by initiating the entire edge simultaneously. In the present examples, a conical charge initiated at the apex was used. Other means of producing a linear shock wave can also be used. If desired, a flat wafer initiated at the center could be employed. If a conical charge is used, the interior of the cone should be filled with an inert material to prevent the formation of a jet which would damage the end of the compact.

In order to reduce noise and air blast from the detonation of the explosive layer, we prefer to submerge the assembly in water prior to initiating the explosive. Inasmuch as the water is not required to transmit the pressure from the detonation of the explosive charge, no confinement of the water is required. The explosive compositions used when the assembly is submerged must obviously be water-resistant. To facilitate recovery of the compact, we prefer to attach a retaining means, such as a cable or chain, to the base plate of the assembly. If the assembly is initiated in air, no retaining means is needed to prevent the compact from being thrown by the blast.

The following example illustrates the compaction of powder in air and also the effect of the explosive loading on the compaction.

Example 22

The procedure described in Example 1 was followed, except that the assemblies were on the ground without cover during the detonation of the explosive layer. The tubes were of cold-drawn seamless steel having an internal diameter of 1½ inches, a wall thickness of ¼ inch and a length of 8 inches. The powder used was electrolytic iron and was vibrator packed to a density of about 3.8 grams per cubic centimeter in the tube.

Tube	Explosive loading (g./sq. in.)	Ratio ¹ of explosive weight to powder weight	Compact diameter (in.)	Compaction ratio
A-----	2	1:11	1½½	1.46:1
B-----	3	1: 7.6	1¾½	1.95:1
C-----	4	1: 6	1¾½	2.2:1

¹ Excluding the conical explosive layer.

The surfaces of all compacts had a smooth metallic

appearance. Compact A had an unconsolidated center $1\frac{3}{32}$ inches in diameter, but the tubular shell of consolidated iron provided excellent rigidity. Compact B had an unconsolidated center $1\frac{1}{16}$ inch in diameter, and compact C had an unconsolidated center $\frac{3}{8}$ inch in diameter. Both compact B and compact C had excellent strength and an average density of more than 90% of theoretical. As may be seen by reference to Example 5, when a similar compaction was made under water of iron powder in a tube having the same diameter and wall thickness, and using 4 grams of explosive per square inch, a density equal to 98% of theoretical was obtained. This higher density is apparently due to the confinement provided by the water surrounding the explosive charge rather than to any transmission of pressure through the water. Thus, when the compaction is made in a denser medium, a lower ratio of explosive to powder can be used than when the compaction is carried out in a less dense medium. The presence of a surrounding relatively dense medium, such as water, also serves to reduce the likelihood of the compact being shattered as a result of rarefaction due to the great difference in the velocity of sound in the two media.

The compacts produced by the method of the present invention have been shown to have considerably higher densities than those obtained by conventional pressing methods. For example, using electrolytic iron containing 1% by weight of zinc stearate as a lubricant, published data indicate the following densities can be expected.

Pressure applied (lb./sq. in.)	Density (g./cc.)
40,000	5.4
50,000	5.8
60,000	6.05
70,000	6.23
80,000	6.37
90,000	6.50
100,000	6.60

As shown by Examples 5 and 13, the densities obtained by the present method are 7.6 grams per cubic centimeter and higher. Published data show that for the same powder at a green (unsintered) density of 6.0 grams per cubic centimeter, a maximum tensile strength of 3800 pounds per square inch can be expected, and for a sintered compact of this density, a maximum strength of 19,000 pounds per square inch can be obtained. A tensile strength specimen was taken from the compact described in Example 13 and a measurement of 13,600 pounds per square inch was obtained. The foregoing shows conclusively the superiority in both density and strength of the method of the present invention over prior methods of compacting powders.

The present method of compacting powders is par-

ticularly applicable to the preparation of compacts of the refractory metals such as titanium and niobium, and for the compacting of powder compositions having hard and/or abrasive particles. Powders which have been compacted by the present method in addition to those shown in the examples include iron oxide, ferrosilicon, sodium chloride, lead dioxide, red lead, alundum and carborundum. As previously indicated, the powders may be compacted in a tube of the same composition as the powders, thus eliminating the need for removal of the tube from the compacted powder. To provide linear strength, wires or rods of the material being compacted may be positioned in the powder prior to compaction; such wires and rods will then reinforce the compact without providing an impurity.

In those cases where the presence of air or other gas may be deleterious to the powder being compacted, the tube assembly may be evacuated prior to detonation of the explosive layer. If only air is objectionable, the air may be replaced with an inert gas. Obviously, a gas which would impart desirable characteristics to the powder may be included in the tube.

If desired, the powder may be heated prior to being compacted. Induction heating can be used to heat powders in the tube, and an explosive having high thermal stability can be used. Similarly, a magnetic field can be impressed on the powder to orient magnetic material in the powder prior to compaction.

The invention has been described in detail in the foregoing. Many modifications and variations that lie within the scope of this invention will occur to those skilled in the art. Accordingly, we intend to be limited only by the following claims.

We claim:

1. A process for compacting powder which comprises surrounding a mass of powder with a layer of high-velocity detonating explosive, said layer being substantially continuous and uniform in composition, and initiating the entire periphery of one end of said explosive layer substantially simultaneously.

2. A process for compacting powder which comprises surrounding a tubular container of a mass of powder with a layer of high-velocity detonating explosive, said explosive layer having a substantially continuous and uniform composition, and positioning a detonating explosive having a conical configuration with the base of said cone adjacent to one edge of said layer of explosive, and thereafter initiating said conical explosive at its apex.

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