

March 15, 1960

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2,928,733

SINTERING OF METAL ELEMENTS

Filed June 21, 1957

2 Sheets-Sheet 1

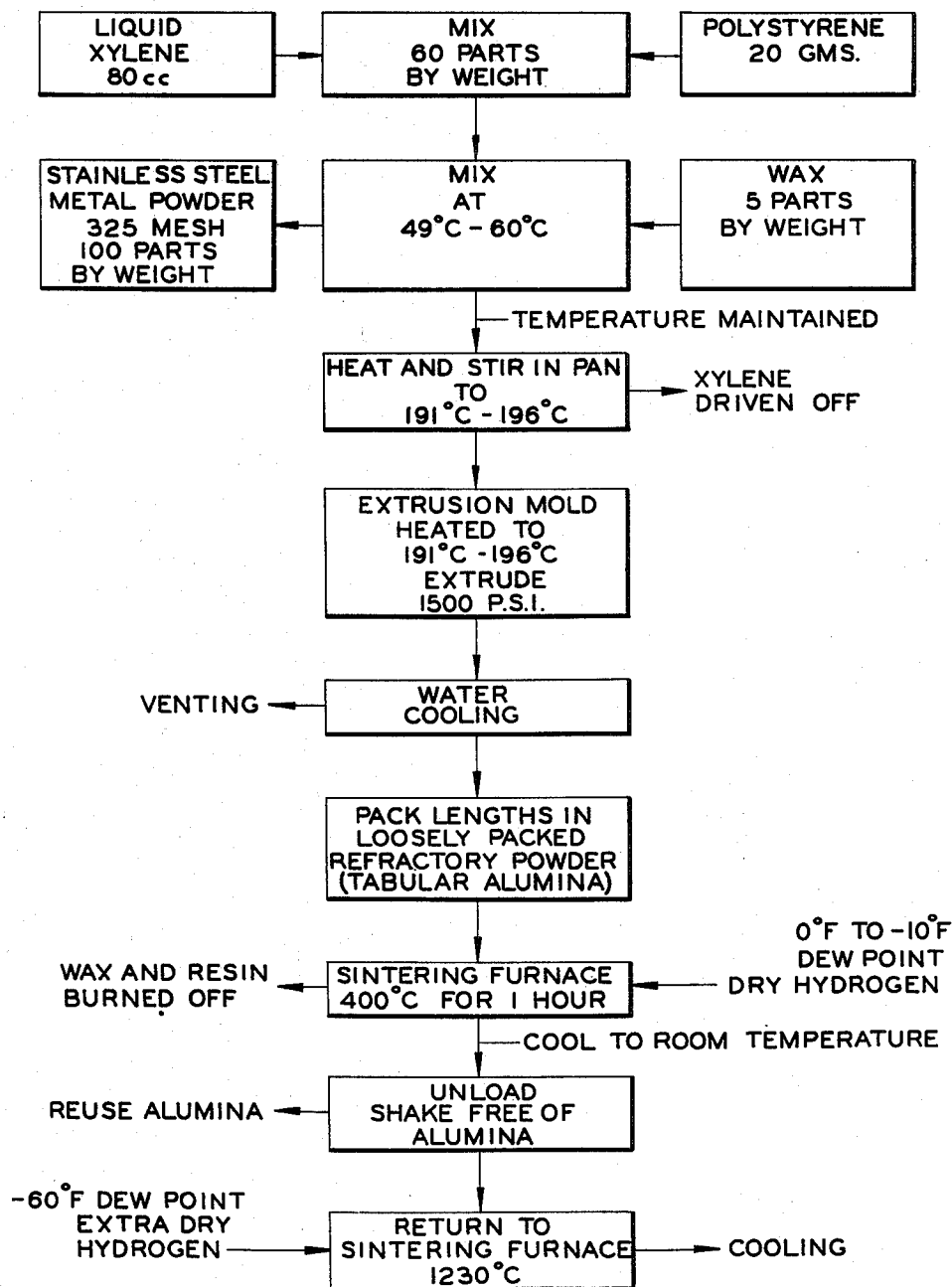


FIG. 1

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2 Sheets-Sheet 2

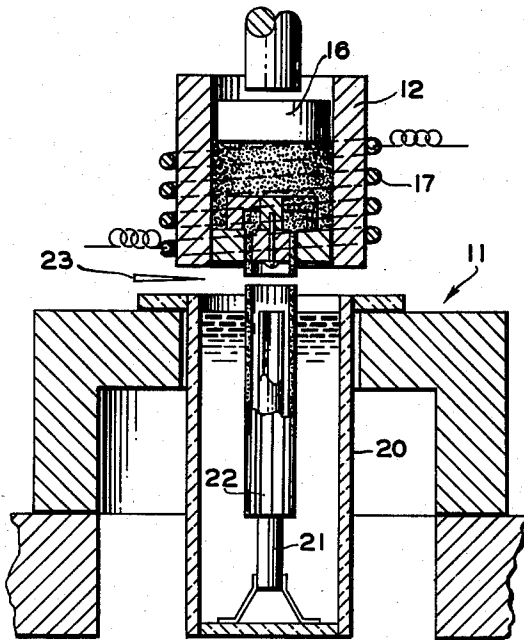


FIG. 4

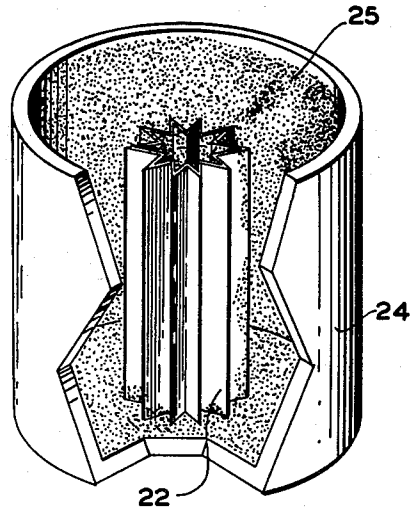


FIG. 5

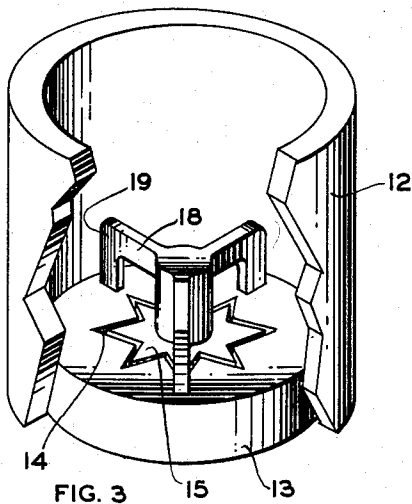


FIG. 3

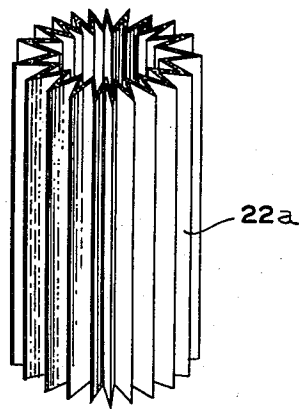


FIG. 2

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SINTERING OF METAL ELEMENTS

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3 Claims. (Cl. 75-221)

This invention relates to improved porous sintered metal elements and methods of making such elements. More particularly it relates to procedure for preparing elements by metal powder extrusion technique and for effectively sintering them.

In the preparation of stainless steel elements stainless steel powder mixed with synthetic resin binder, organic solvent and a lubricant are extruded, for example, as tubing having convoluted section. The extruded tubing is then cut into "green" lengths and subjected to sintering procedure.

During sintering these elements require support because of the softening and burning off of the resin binder at low temperature, which would result in collapse of unsupported elements. To prevent this collapse, the elements are supported by embedding them in a setter of loosely packed refractory powder prior to elevation to sintering temperature.

Experimental research has shown that the use of proper setter materials is absolutely essential because improper setter materials are found to react chemically with the metal of the "green" element or to cake or to adhere so strongly to the "green" element after sintering as to render it expensive and difficult to produce clean setter free sintered elements.

Objects and features of the present invention are the provision of satisfactory setter material for sintering operations and novel sintering procedure using such material to provide minimal adherence of setter material to the sintered elements and ready removal of the small amount retained by simple mechanical brushing or the like.

Other objects and features of the invention will become apparent from the following description and accompanying drawings, wherein:

Figure 1 represents a flow diagram of a preferred method of practicing the invention;

Figure 2 illustrates in perspective a filter element prepared in accord with the method;

Figure 3 is a perspective, partially broken away view of a die used in extruding the element of Figure 2;

Figure 4 is a sectional elevation of extruding apparatus using the die of Figure 3; and

Figure 5 is a perspective view partially in section of an extruded "green" element packed in setter material for sintering operations.

In a specific example of practicing the invention stainless steel powder of minus 325 mesh is used. A preferred powder is 18Cr-8Ni type with the addition of about 2.5% silicon which latter permits sintering to occur at temperatures below 1260° C. whereas non-silicon containing powders require at least 1315° C.

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While minus 325 mesh stainless steel powder is preferred, powders of other dimensions are contemplated as useful. Likewise other steel, bronze, brass, titanium, zirconium, aluminum and other metal powders may be used.

The composition of the preferred stainless steel is as follows:

	Percent
C -----	0.10
Si -----	2.37
Mn -----	0.85
S -----	0.008
Cr -----	18.11
Ni -----	8.19
Fe -----	Balance

Although as stated a wide range of particle sizes can be used, in accordance with the porosity required, a typical powder useful in practicing the invention comprises particles in the 100 to 325 mesh range.

In preparing the powder for extrusion, it is found desirable to mix it with material that will assist in shaping it to final form and retain it in such form as a "green" element after extrusion. Accordingly, a heat depolymerizable organic binder is dissolved in a volatile aromatic hydrocarbon solvent and mixed with the powder. A typically useful binder is polystyrene which is capable of being driven off during heating without leaving carbonaceous, tarry or gummy residues that would char during sintering.

The polystyrene is mixed with the solvent which may be xylene, at room temperature in the ratio of 20 grams of resin to 80 cc. of xylene. This forms a fairly viscous liquid to which is added 100 parts by weight of minus 325 mesh stainless steel powder per 60 parts by weight of the liquid.

This mix is stirred thoroughly and warmed to 49-60° C. At this time 5 grams of a lubricant wax, for example, Cardis No. 319 die wax (a product of Warwick Wax Company, Long Island City, New York) is melted and added to the mix which is then transferred to a pan. Further heating and stirring of the mix in the pan is effected at a temperature of from 191-196° C. resulting in uniform distribution of the wax and driving off of more xylene until a plastic mass results having a putty like consistency. The extruding apparatus is maintained in the range of extrusion temperature, namely, 191-196° C.

The mass is now ready for extruding and is transferred to the extruding apparatus 11 of Figure 4. This apparatus 11 forms no part of the instant invention but is merely illustrative of the types of devices that can be used.

The heated mixture at 191-196° C. is placed in a heated die cylinder 12 which has a base 13 having a convoluted section outlet 14 in which a convoluted die 15 is mounted. A ram 16 at the upper end of cylinder 12 is operated by a power source (not shown) in order to force the heated mixture mass through the limited convoluted opening 14 in base 13 past die 15. An electric heater coil 17 is positioned around cylinder 12 for maintaining the temperature of the mass mixture at from 191-196° C.

The die cylinder 12 and its die 15 are more fully shown in Figure 3. The die 15 as an insert is fastened to a spider 18 whose legs 19 are secured to the die base 13 so as to support the insert concentrically within the base opening 14. In the embodiment shown both the opening

14 and the insert 15 have convoluted section and are concentrically spaced from each other.

The die cylinder 12 is installed as shown in Figure 4 so that the heated mass from cylinder 12 is extruded by ram 16 via the annular space defined between insert 15 and opening 14. The head pressure created by the ram is in the range of approximately 1500 p.s.i. This pressure is a variable factor and may range from 100 p.s.i. upwardly depending on the desired porosity. It must be sufficiently high to fill the die space with extrudant mass yet not be so great that the metal particles become too tightly packed during extrusion.

Cooling of the extrusion can be effected directly as it leaves the exit end of the die to prevent collapse by direct extrusion into water contained, for example, in a water tank or jacket 20 which has a removable extrusion receiving core 21 onto which the hollow extrusion leaving the die base 13 and severed into prescribed lengths is received.

When the extruded tube leaves the exit end of die base 13, it slides onto the core 21 in the water tank 20 moving thereover until cut into desired "green" units 22 of selected length, for example, by a hot wire or by reciprocating cutter 23 and each "green" unit 22 is now ready for the next step in the process. It is to be distinctly understood that the specific apparatus of Figures 3 and 4 may be replaced by any other type of extrusion apparatus that will yield comparable "green" units 22 of the type described. For example, the apparatus of co-pending Kovacs and Wainer application Serial No. 515,010, filed June 13, 1955, and now abandoned, may be used. Moreover, "green" units having other configurations may be similarly provided.

At this point in the process the solvent xylene has all been driven off and the metal powder particles are held together in a "green" or unconnected state by the resin and wax so that it is necessary to provide special support for them during sintering steps whose heat first melts them then burns away the resin and wax.

In order to provide such support each "green" unit 22 is placed in a container 24 in which it is completely packed in an inert refractory powder preferably powdered alumina oxide. A satisfactory grade of such alumina is Tabular Alumina T-61 Grade, minus 60 mesh produced by Aluminum Company of America. This packing 25 is placed inside as well as outside of the "green" element 22 in the container 24 to provide support for all convolutions but is packed loosely enough to prevent cracking due to shrinkage of the "green" units during sintering and also to permit free circulation of gas to the units as will be described.

A container packed as described is then placed in a batch type hydrogen furnace of conventional design whose details form no part of the invention. The furnace is slowly heated to approximately 400° C. during which polystyrene binder and wax are destroyed and vented. Comparatively dry hydrogen having a dew point from 0° F. to minus 10° F. is circulated through the furnace at this time. A yellowish sooty flame occurs during this period. When all binder and wax are driven off the characteristic blue hydrogen flame appears. This takes approximately 1 hour. The container is then cooled in the furnace to room temperature and the partially sintered element 22 removed therefrom and shaken free of the alumina refractory powder which does not stick to the unit 22 and placed in an unloaded or fresh container and returned to the hydrogen furnace for a second sintering stage.

The furnace now is elevated to a sintering temperature of about 1230° C. and extra dry hydrogen having a dew point of approximately minus 60° F. is circulated through the furnace and in contact with the unsupported element 22 therein. This second sintering stage or operation is carried on from 1½ to 4 hours and effectively sinters the steel powder particles together into a strong element.

Thereafter the element is cooled to room temperature in the furnace and removed. If by chance some residual alumina refractory is found clinging to the sintered element, it may be removed by tumbling in a tumbling barrel containing hot dilute sodium hydroxide (NaOH) solution long enough to dislodge such particles and otherwise clean the element.

The elements are then washed, air dried and ready for use.

The dryness of the hydrogen used during the two sintering steps is critical in obtaining good strength in the sintered elements particularly since high pressure is not used during extrusion. Since the extrusion pressure neither deforms the powder particles nor forces intimate contact between them, all particles being enveloped in a thin film of resin, oxide films on the particles are not ruptured during extrusion. Hence the moisture content of the hydrogen is critical to prevent sticking. When the first sintering step occurs the hydrogen (0° to -10° F. dew point) is moister than that used in the second sintering step (-60° F. dew point hydrogen). This moister hydrogen prevents the alumina from sticking to the preliminarily sintered element.

Inasmuch as the alumina is shaken away from the preliminarily sintered element before the second stage of sintering with dry hydrogen (-60° F. dew point) is effected, the problem of clinging alumina is substantially eliminated and great strength can be imparted to the element by second stage sintering in the extra dry hydrogen for periods up to 4 hours.

Should some residual alumina powder be found clinging to the second stage sintered elements, the dilute acid leaching described can be employed if stiff brushing will not remove it. In the alternative, steam blasting or sodium hydride oxide reduction can be used to rid the sintered elements of such residual refractory.

Porosity of the product is controllable by regulation of particle size, proportion of powder and binder temperature and time of sintering.

While a preferred embodiment of the invention has been described and shown, variations within the scope of the appended claims are possible and are contemplated. There is no intention, therefore, of limitation to the exact disclosure herein made.

What is claimed is:

1. In a process of preparing a sintered metal element from a "green" unit of extruded metal powder-organic binder mix, the steps comprising supporting the "green" unit in refractory alumina powder and effecting first stage sintering of the powder supported unit at a temperature of approximately 400° C. in an atmosphere of dry hydrogen whose dew point ranges from 0° to -10° F. during which the organic binder is burned off, then removing the unit from the alumina powder support, ridding it of clinging alumina powder and thereafter effecting second stage sintering of the unsupported unit at a temperature of about 1230° C. in an atmosphere of extra dry hydrogen whose dew point approximates -60° F. for a period of from 1½ to 4 hours.

2. In a process of preparing a stainless steel sintered element from a "green" unit of extruded stainless steel powder-organic binder mix, the steps comprising supporting the "green" unit in refractory alumina powder and effecting first stage sintering of the powder supported unit at a temperature of approximately 400° C. for about one hour in an atmosphere of dry hydrogen whose dew point ranges from 0° to -10° F. during which the organic binder is burned off, then removing the unit from the alumina powder support, ridding it of clinging alumina powder and thereafter effecting second stage sintering of the unsupported unit at a temperature of approximately 1230° C. for a period of from 1½ to 4 hours in an atmosphere of extra dry hydrogen whose dew point approximates -60° F.

3. A process of preparing sintered metal elements comprising the steps of extruding at a pressure of between 100 p.s.i. and approximately 1500 p.s.i. a "green" element of powdered metal and organic binder into desired shape, packing the "green" element in a refractory powder support, effecting first stage sintering of the powder packed element in an atmosphere of dry hydrogen having a dew point of from 0° to -10° F., removing the first stage sintered element from its powder packing, ridding it of clinging powder packing, and effecting second stage sintering of the first stage sintered element in an

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extra dry hydrogen atmosphere whose dew point approximates -60° F.

References Cited in the file of this patent

UNITED STATES PATENTS

1,008,806	Farkas	Nov. 14, 1911
1,922,038	Hardy	Aug. 15, 1933
2,152,006	Welch	Mar. 28, 1939
2,471,630	Kurtz	May 31, 1949
2,719,786	Fredenburgh	Oct. 4, 1955