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E. D. FISHER

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ALUMINUM FILTERS AND METHOD OF PRODUCTION

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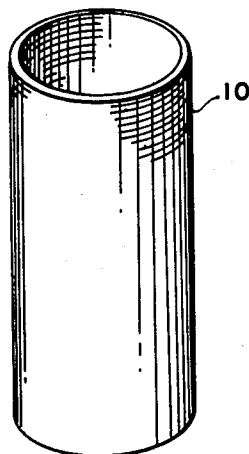
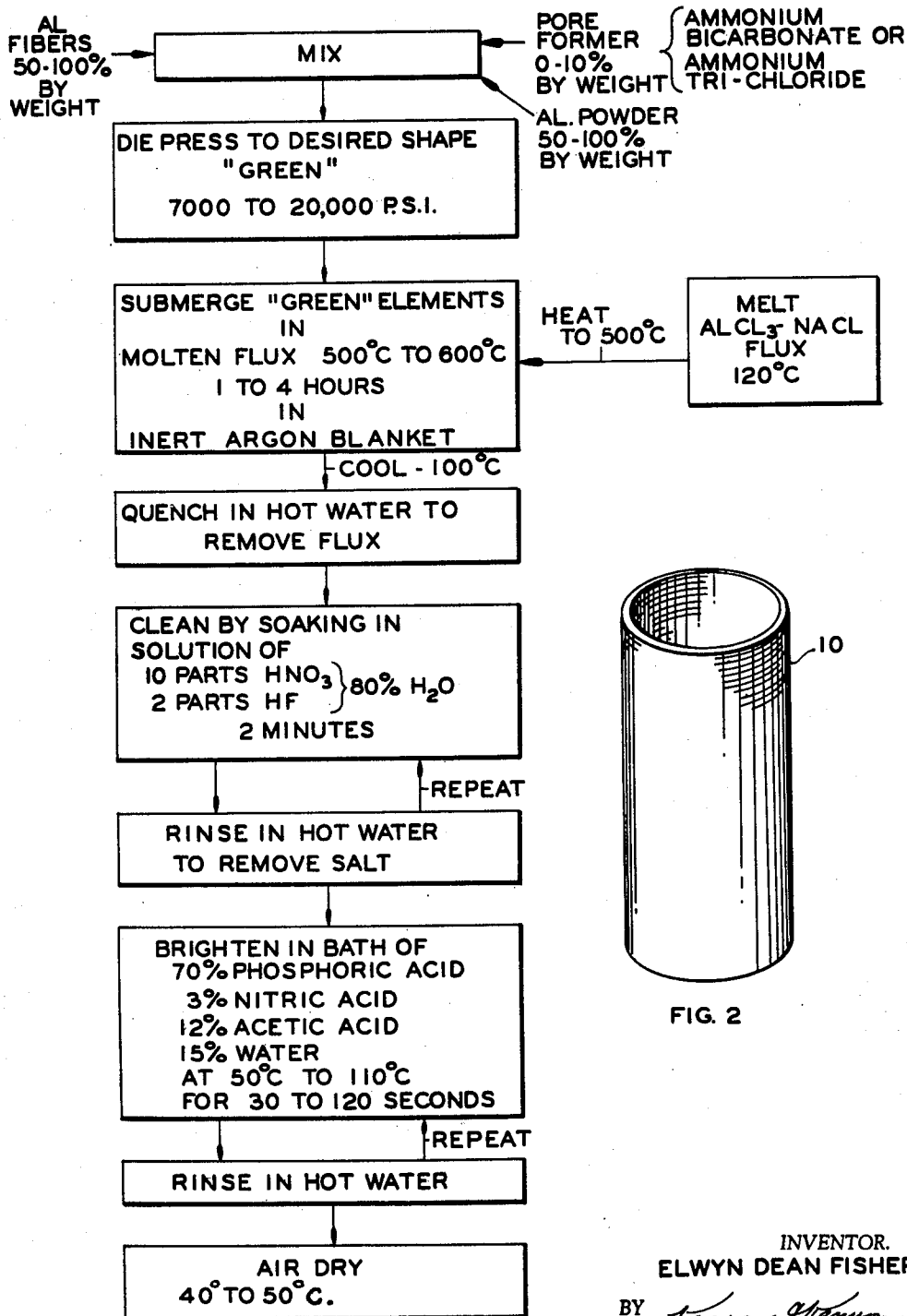


FIG. 2

FIG. 1

INVENTOR.
ELWYN DEAN FISHER

BY *Kenyon & Kenyon*
ATTORNEYS

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ALUMINUM FILTERS AND METHOD OF PRODUCTION

Elwyn Dean Fisher, Cleveland, Ohio, assignor to Puro-lator Products, Inc., Rahway, N. J., a corporation of Delaware

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5 Claims. (Cl. 75—223)

This invention relates to improved metallic elements and more particularly to elements of this type made of sintered aluminum or aluminum base metals.

In the preparation of porous metallic elements from aluminum fibers or powder and other readily oxidized powdered metals, "green" compacts of the powdered metals pressed to desired shape require a sintering treatment to produce strong final elements having the form created in the "green" compacts. The sintering treatment of aluminum and other readily oxidized materials, however, is complicated by the film of oxide on the particles or fibers which interferes with metal to metal bond between the particles or fibers. This interference ordinarily results in unsatisfactory fragile elements that have no commercial utility. It is necessary to provide special process features to eliminate the oxide film and to permit sintering to occur.

Principal features and objects of the present invention are the provision of a sintering treatment for aluminum elements which will achieve sufficient removal of the oxide film and permit the required effective sintering to occur.

Other objects and features of the invention are the provision of effectively sintered porous aluminum metal elements that are strong, have satisfactory flow rate and relatively low pressure drop as well as controllable pore size, that may be used for filters, heat exchangers, bearings and the like.

Further objects and features of the invention are the provision of practical methods of preparing commercially useful elements of sintered aluminum fiber or powder particles for use as filters, heat exchange or heat transfer elements, bearings or for other general uses.

In practicing the invention a mass of metallic aluminum particles such as fibers or powders or combinations of both with or without added pore or void formers such as ammonium bicarbonate, aluminum trichloride or the like, are given a desired "green" shape by pressure for example in a mold or die. In this pressing action, the oxide film on the particles is ruptured, bringing clean metal in adjacent particles into contact. Then the shaped "green" elements are submerged in molten flux heated to the sintering temperature of the aluminum for a sufficient length of time to remove oxide film and keep the metal surfaces clean and free of exposure to oxygen and to permit sintering junction of the particles to occur while in the heated flux. Thereafter the sintered elements are removed from the flux, washed to rid them of flux and then cleaned in an acid bath and brightened in a brightening bath. After final washing the sintered, cleaned and brightened elements are dried and ready for use.

Additionally, the invention contemplates the use of other readily oxidizable metal particles alone or in combination with aluminum for the production of sintered filter elements. In such practice the flux selected for use during sintering of the "green" elements is selected to be compatible with the metal or metals used and effective in removing the oxide film and allowing effective sintering to occur.

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Additional objects and features of the invention will become apparent from the following description and the accompanying drawing wherein:

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

Figure 2 illustrates in perspective an aluminum filter element prepared in accordance with said method.

Referring to the drawing, the reference numeral 10 denotes a porous sintered aluminum filter element prepared in accord with the process of this invention.

In a specific example of practicing the invention, aluminum metal fibers of the order of $\frac{1}{4}$ " to $\frac{3}{8}$ " long 200 mesh diameter and aluminum powder of the order of 200 mesh screen size preferably in equal parts by weight are mixed together thoroughly with the addition of up to 10% by weight of a void or pore former such as ammonium bicarbonate or ammonium trichloride.

The mixture is placed, for example, in a cylindrical die lubricated, for example, by lithium or calcium stearate and compressed under a pressure of from approximately 6,000–20,000 p. s. i. to produce cylinders called "green" compacts. Other desired "green" compact shapes may be effected by such compression. The high pressure used is believed to rupture the oxide film and allow contact of clean metal in adjacent particles and also permit ready removal of the oxide by an appropriate flux in sintering operations. Sintering steps are now effected on the "green" compacts in the presence of an appropriate aluminum flux.

A preferred flux found useful for sintering purposes is a sodium chloride-aluminum trichloride flux of the type described in Wagner U. S. Patent 2,723,929, dated November 15, 1955, and preferably consisting of a homogeneous mixture of from 70%–90% by weight of aluminum trichloride and 30%–10% by weight of sodium or other alkali metal chloride. This flux is melted at approximately 120° C. becoming a clear liquid and is then heated to an aluminum sintering temperature of from 500° to 600° C.

The "green" compacts are deposited and submerged in the clear and heated molten flux bath over which an inert blanketing atmosphere as of argon is maintained and are maintained therein at the sintering temperature of from 500° to 600° C. for a period of approximately 1 to 4 hours during which sintering of the aluminum particles and their junction by sintering occurs. The flux described has been found effective. Other fluxes and atmospheres are contemplated as useful in practicing the invention.

Upon completion of the sintering step, the sintered compacts in the form of tubes are removed from the flux bath in the inert atmosphere, for example, argon and cooled to about 100° C. They then are quenched in hot water and allowed to soak therein to remove flux and, if present, the pore forming ammonium bicarbonate from the pores of the sintered tubes. This soaking is carried out long enough to leach the major portion of the flux and other materials from the pores.

Following this water leaching step, the tubes are cleaned, for example, by soaking in a solution of 10 parts of nitric acid HNO_3 and 2 parts of hydrofluoric acid HF and 80% water for about two minutes. This acid soaking is followed immediately by a hot water rinse and the acid soaking and rinsing steps are alternated until all salt has been removed from the sintered elements.

Thereafter, the acid soaked, water rinsed, clean elements are brightened in a brightening bath maintained at a temperature of from 50–110° C. into which they are dipped for 30–120 seconds at a time, followed immediately by a hot water rinse repeated alternately until the surfaces of the tubular elements are bright. The

brightener bath in a preferred embodiment consists by volume of:

- 70% phosphoric acid
- 3% nitric acid
- 12% acetic acid
- 15% water

Other appropriate brightener baths may be used.

The thinner the walls of the elements the faster the brightener bath acts and consequently its temperature and the time of dipping therein respectively must be lower and shorter. Careful correlation to wall thickness is necessary.

After a final hot water rinse, the cleaned and brightened, sintered, tubular, aluminum filter elements are dried in air or in an oven at a slightly elevated temperature ranging from 40–50° C.

The dried products resulting are the desired tubular filter elements of this invention.

Although the invention has been described specifically with reference to aluminum elements, other readily oxidizable metals can be processed similarly by the use of appropriate fluxes that will rid them of their oxide films during sintering operations.

The porosity and other qualities of these elements depend on a number of variables, namely:

- (a) Proportion of powder and pore former
- (b) Size and type of powder particles
- (c) Temperature of sintering steps
- (d) Duration of sintering process steps
- (e) Wall thickness of the elements

Variations in any of these factors may be used as controls to vary porosity of the elements, rate of flow throughout, strength and length of life as well as other qualities of the filter elements produced.

An alternative procedure for producing aluminum elements of desired shape is to prepare a molten bath of the hereindescribed flux, then cool this bath to viscous state and add to it particles of aluminum powder or fibers or a mixture of these two providing a plastic mass, then shaping the plastic mass to desired "green" form of the elements as by pressure or extrusion, and thereafter heating the "green" formed elements, for example, in an inert argon atmosphere to a sintering temperature of from 500°–600° C. Such procedure will permit production of sintered simple strip or block-like shapes. With tubular or convoluted shapes, however, provision must be made for support of the shaped elements during sintering to prevent disintegration. Such support may, for example, be provided by packing the "green" forms with a refractory material during sintering.

Still another alternative procedure comprises mixing particles of aluminum powder alone or fibers alone or mixtures of these two with sodium chloride and hot pressing such mixture into desired shape at temperatures of from 385°–400° C. for a period of time sufficient to effect sintering union of the aluminum particles and/or fibers. The temperature and pressure in relation to time will effect the extent of sintering effected in the final product.

While, therefore, preferred methods of practicing the invention have been described, variations within the scope of the appended claims are possible and are contemplated. There is no intention, therefore, of limitation to the exact details shown and described.

What is claimed is:

1. A process of preparing sintered aluminum elements

comprising the steps of compressing a mixture of aluminum particles of small size and of an aluminum pore former compound, into desired shapes thereby forming "green" compacts, providing an aluminum flux consisting of an intimate homogeneous mixture of 70%–90% by weight of aluminum trichloride and 30%–10% by weight of alkali metal chloride, melting this flux and heating the molten flux to an aluminum sintering temperature of from 500°–600° C., submerging the "green" compacts in this molten and heated flux for a period of from 1–4 hours, thereafter removing said compacts from said bath as sintered aluminum elements and then cleaning and brightening said elements.

2. A process of preparing sintered aluminum elements comprising the steps of compressing aluminum particles into desired shapes forming "green" compacts, providing a molten aluminum flux bath comprising aluminum chloride and alkali metal chloride, heating the bath to an aluminum sintering temperature of from 500°–600° C., submerging the "green" compacts in said molten heated flux bath for a period of from 1 to 4 hours to effect sintering union of the aluminum particles in the "green" compacts to form sintered aluminum elements, thereafter removing the elements from the bath, leaching the same with water to rid them of retained flux, then acid cleaning the leached elements, and, thereafter brightening the acid cleaned elements in a brightener bath.

3. The process of claim 2 wherein said brightener bath comprises a solution containing approximately 70% phosphoric acid, 3% nitric acid, 12% acetic acid and 15% water.

4. The process of claim 2 wherein the acid cleaning is effected in a bath which consists of approximately 10 parts of nitric acid HNO_3 and 2 parts of hydrofluoric acid HF mixed with water in the proportion of 80% water and 20% nitric acid hydrofluoric acid mix.

5. A process of preparing sintered aluminum elements comprising the steps of providing aluminum fibers and aluminum powder, adding a pore former to form a mixture, compressing the mixture at a pressure ranging from 6,000 to 20,000 p. s. i. to form "green" compacts, providing a molten aluminum flux bath, elevating the temperature of the molten flux bath to from 500°–600° C., submerging the "green" compacts in this molten flux bath at said temperature for a period of from 1 to 4 hours to effect sintering junction of the aluminum fibers and powder, then removing the compacts from the molten flux bath, then leaching the compacts with water to rid them of retained flux and pore former, thereafter effecting acid cleaning of the leached compacts in an acid cleaning bath comprising nitric acid, hydrofluoric acid and water and thereafter brightening the cleaned compacts in a brightener bath comprising a mixture of phosphoric acid, nitric acid, acetic acid and water.

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