

NO DRAWINGS

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(54) METHOD OF GROWING ALPHA-ALUMINA WHISKERS

(71) We, GENERAL TECHNOLOGIES CORPORATION, a Corporation organised under the laws of the State of Delaware, United States of America, of 1821, Michael Faraday Drive, Reston, Virginia, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a method of growing single-crystal alpha-alumina whiskers or fibers.

Single crystal whiskers or fibers of metals and metallic compounds possess unusually high strengths as compared to the bulk material so that the whiskers can serve as reinforcement materials for structural elements, alpha-alumina whiskers being suitable as one such material.

Patent Specification No. (Application No. 7003/68) Series No. 1,203,342 discloses a method of growing a mat article comprising interconnected groups of single crystal alpha-alumina fibers, including the step of passing hydrogen and water vapour over a charge of molten aluminum in a ceramic receptacle provided with a loosely-fitting cover of ceramic material so that deposition of said fibers occurs at spaced-apart sites on the underside of said cover thereby to form en-meshed groups of said fibers wherein the fibers in each of said groups are connected together substantially at and radiate from a respective one of said sites. There are also other methods in which hydrogen gas containing water is passed over molten aluminum and some of the aluminum is oxidized thereby to form alpha-alumina whiskers.

The present invention is intended to provide an improved method of growing alpha-alumina whiskers with a view to obtaining a high yield and good crystalline formation.

According to the invention there is employed a method of growing single-crystal alpha-alumina whiskers including the step

of passing a gas and water vapour mixture over a charge of molten aluminum in a ceramic receptacle at an elevated temperature, characterized in that the said gas is hydrocarbon gas. 50

Except that hydrocarbon gas is used as a reagent atmosphere, methods substantially as used previously for making alpha-alumina whiskers may be employed. Accordingly an aluminum charge in a refractory receptacle is heated to an elevated temperature in a protective atmosphere, and then dry gas is passed over the molten charge in order to reduce the surface tension of the charge in the receptacle. 60

Then in accordance with the present invention mixture of water vapour and hydrocarbon gas, for example saturated hydrocarbon gas or unsaturated hydrocarbon or natural gas or a mixture thereof, is passed over the charge so that as the hydrocarbon gas contains water it causes alpha-alumina whiskers to be formed in the reaction chamber. The whiskers usually grow over the charge and on the sides of the receptacle. 70

The flow rate of the hydrocarbon gas is preferably maintained at between 0.0002 and 0.0007 cu.ft./sec., at a temperature of between 2400° and 3000°F during whisker growth. 75

The water content of the hydrocarbon gas is advantageously maintained between limits of 500 p.p.m. and 3000 p.p.m., and preferably the water content is increased gradually during the whisker growth period from near the lower limit to near the higher limit. 80

The following Examples further describe how the invention may be performed. 85

EXAMPLE I

A receptacle for the aluminum charge is prepared by mixing 3 lbs. 2 oz. of ceramic powder having the following composition: 75 parts alumina, 15 parts silica, 5 parts titania, 2.5 parts ferric oxide and 2.5 parts other metallic oxides (by weight), and 0.16 lbs. of 200 mesh aluminum powder (i.e. powder of 90

particles small enough to pass through a mesh with apertures each $1/100$ inch \times $1/100$ inch in size), being 5% by weight of aluminum. The receptacle is fired in air at 2200°F. for about a half-hour, and its internal dimensions are 5 inches wide \times 17 inches long \times 2 inches deep. A charge of 2 lbs. 8 oz. of aluminum pellets is spread evenly over the bottom of the receptacle, which is loosely fitted with a cover substantially of the same composition as that of the receptacle except that the powdered aluminum is omitted. The cover is prepared by mixing the constituents and shaping and then firing the ceramic in air at 2200°F. for about a half-hour. The charged receptacle is inserted into a quartz tube having a cross-sectional area of about 20 sq. inches. The effective open area of the tube is about 12 square inches. The tube is placed in a furnace set at 2550°F. Thus the apparatus and method employed in this Example are in some respects similar to those described in Patent Specification No. (Application No. 7003/68) (Serial No. 1,203,342).

The surface tension of the molten aluminum charge is reduced by passing a protective stream of dry hydrogen gas (less than 20 p.p.m. water) over the charge for about 10 minutes. Then the flow of dry hydrogen gas is shut off and wet hydrocarbon gas is admitted. Growth of whiskers then begins. The hydrocarbon gas contains mostly methane (93.6%) and ethane (3.71%), plus smaller amounts of other higher hydrocarbons. The water content of the gas is about 500 p.p.m. The flow rate is 0.0007 cu.ft./sec. After 15 minutes the water content of the gas is increased to 3000 p.p.m., the whisker growth period lasts 45 minutes.

Alpha-alumina whiskers are obtained principally on the underside of the cover in the form of a mat article. The mat article is about $1/16$ " thick, and contains whiskers each having a diameter of between 1 and 3 microns. The density of the mat article is about 0.01 g./c.c. and the yield is 1.0 g.

EXAMPLE II

The flow rate of methane gas during a procedure otherwise substantially as described in Example I is decreased to 0.00025 cu.ft./sec., and the furnace temperature is increased to 2700°F. This increases the yield of whiskers and the diameter of the whiskers produced. The yield of alpha-alumina is 2.8

g. and the diameter of the whiskers is between 1 and 10 microns. The density of the mat article produced is about 0.02 g./c.c.

EXAMPLE III

During a procedure otherwise substantially as described in Example II the temperature is lowered to 2600°F. The yield is 2.1 grams of an alpha-alumina mat article of whiskers having diameters between 1 and 6 microns, with a density of 0.02 g./c.c.

In view of Section 9 of the Patents Act 1949 attention is directed to Patent No. (Application No. 7003/68) (Serial No. 1,203,342).

WHAT WE CLAIM IS:—

1. A method of growing single-crystal alpha-alumina whiskers including the step of passing a gas and water vapour mixture over a charge of molten aluminium in a ceramic receptacle at an elevated temperature, characterized in that the said gas is hydrocarbon gas.

2. A method according to Claim 1, characterized in that the hydrocarbon gas is a saturated hydrocarbon gas or an unsaturated hydrocarbon gas or a natural gas or a mixture thereof.

3. A method according to Claim 1 or 2, characterized in that the flow rate of said hydrocarbon gas is maintained at between 0.0002 and 0.0007 cu. ft./sec.

4. A method according to Claim 3, wherein the temperature is maintained between 2,400°F. and 3,000°F. during whisker growth.

5. A method according to Claim 3 or 4, wherein the water content of the hydrocarbon gas is maintained between limits of 500 p.p.m. and 3,000 p.p.m.

6. A method according to Claim 5, wherein the water content is increased gradually during the whisker growth period from near the lower limit to near the higher limit.

7. A method of growing whiskers of alpha-alumina substantially as described in any of the Examples hereinbefore.

8. Whiskers of alpha-alumina when grown by a method according to any of the preceding Claims.

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