METHOD OF PREPARING P_2O_5-SiO_2 PRODUCTS

THE INVENTION

The present invention relates to a process for forming oxide products of phosphorus and silicon and more particularly, the present invention relates to the formation of phosphorus pentoxide-silica containing glass compositions from gelled polymers having phosphorus-oxygen-silicon linkages.

In my copending application, U.S. Ser. No. 132,581, there is described a process for forming homogeneous high purity oxide products of phosphorus and silicon, this application being hereby incorporated by reference. In that application, there is disclosed a method for forming oxide products of phosphorus and silicon having phosphorus-oxygen-silicon linkages, wherein a silicon alkoxide is first, at least partially hydrolyzed with water in the presence of an effective catalytic amount of hydrolysis catalyst to produce a single phase solution of an at least partially hydrolyzed silicon alkoxide, which material is then reacted with phosphoric acid, phosphorous acid, phosphorus pentoxide or mixtures thereof to form a clear solution of a soluble, substantially linear, further polymerizable polymer having phosphorus-oxygen-silicon linkages and then this soluble, substantially linear polymer is converted to a cross-linked polymer in the presence of a sufficient quantity of water to form a gel of said solution, then the gel is decomposed and an oxide product of phosphorus and silicon isolated and recovered therefrom. When formed into a substantially unitary article, this product represents an excellent sputtering target for use in conventional sputtering applications to form high quality, uniform film on, for example, platinum and silicon substrates. The present application represents an improvement in that process wherein the initial step of at least partially hydrolyzing the silicon alkoxide in the presence of an effective catalytic amount of a hydrolysis catalyst may be omitted.

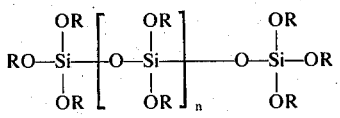
Thus, it has been surprisingly found that there is no need to employ this extra step nor to add one of the conventional hydrolysis catalysts for example acids; the phosphoric acid, phosphorous acid, or phosphorus pentoxide can now be directly reacted with the silicon alkoxide and in a manner similar to that disclosed in the above-mentioned application, an oxide product of phosphorus and silicon recovered. Thus, by adjusting the amounts of equivalent P_2O_5 added, either in the form of phosphorus pentoxide or the equivalent thereof in the form of phosphorous acid or phosphoric acid relative to the equivalent amount of silica added in the form of silicon alkoxide, excellent substantially unitary, solid bodies or sputterable targets can be produced consisting essentially of about 8 to 26 weight percent P_2O_5, preferably about 15 to 19 weight percent P_2O_5 and about 74 to 92 weight percent SiO_2, preferably about 85 to 81 weight percent SiO_2 and wherein the alkali metal oxide impurities are less than about 300 parts by weight per million.

These substantially unitary solid bodies or targets may be used in any of the conventional sputtering techniques for forming high quality films on various substrates. As used herein, sputtering comprehends any process whereby particles, whether molecular and/or atomic size, are disintegrated, dislodged, evaporated or otherwise removed from a so-called target material and transferred to a selected substrate surface. The removal

of the target material is by means of any appropriate energy, such as electron beam, laser beam, plasma discharge or the like. In a preferred type of sputtering process, the target material is disintegrated and transferred to a selected substrate surface such as, for example, a semiconductive surface by means of an RF sputtering process, that is, a process involving the atomic disintegration of a solid target appropriately struck by ions or atoms in a gaseous plasma discharge. The oxide products of phosphorus and silicon, as produced by the method described herein, are also useful as batch ingredients in conventional batch-forming techniques. This will be found to be particularly advantageous inasmuch as the bonding of the phosphorus-oxygen-silicon linkages in this material show excellent thermal stability and thus volatilization problems and toxicity problems, which are common using conventional materials for supplying P_2O_5 to a silicate glass, will be substantially minimized, if not entirely reduced.

Thus, according to one aspect of this invention, there is provided a method for producing an oxide product of phosphorus and silicon, which comprises: forming or providing a single phase solution intimately contacting phosphoric acid, phosphorus pentoxide, or mixture thereof with a compound represented by Formula I or II below:

I.



$$Si—X_nY_{(4-n)} \qquad \text{(II)}$$

wherein in Formula I, R is C_1 to C_6 alkyl, such as, for example, methyl, ethyl, propyl, butyl, pentyl, hexyl and isomeric forms thereof and n is an integer between 0 to 10, and wherein in Formula II, n is 0 or 1, X is phenyl or a C_1 to C_6 alkyl, such as, for example, methyl or ethyl, and Y is an OR, wherein R is a C_1 to C_6 alkyl, such as, for example, ethyl; adding a sufficient quantity of water to said solution to form a two-phase system; and then removing free liquid components from said two-phase system and thermally decomposing the residue to obtain the oxide product of phosphorus and silicon.

According to another aspect of this invention, a homogeneous oxide product of phosphorus and silicon is obtained by a method which comprises: intimately contacting phosphorus pentoxide, phosphoric acid, phosphorous acid, or mixtures thereof, with (*a*) a compound represented by Formula II above, wherein n is 1 or (*b*) a compound of Formula II set forth above, wherein n is 0, in the presence of an effective solution forming amount of an organic solvent; or (*c*) a compound of the formula represented by I above in the presence of an effective solution forming amount of an organic solvent so as to form a clear solution; converting the solution product produced by the intimate contact above into a homogeneous gel in the presence of an effective gelling amount of water and recovering a homogeneous oxide product from the gel by thermally decomposing it.

When a compound of Formula II is employed, wherein n is 1, such as, for example, methyl triethoxysi-