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DIAMOND ABRASIVE ARTICLE AND
METHOD OF MAKING THE SAMEReinhold A. Schaefer, Rocky Hill, Conn., assignor
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This invention relates to vitrified-bonded diamond abrasive articles such as grinding wheels in which the abrasive element comprises diamond abrasive particles, and to a method of making the same. This application is a continuation in part of my copending application Serial No. 604,979, filed July 13, 1945, and now abandoned.

Among the features and objects of this invention are the provision of a vitrifiable bond of a firing or maturing temperature below that at which oxidation of the small diamond particles takes place so that the firing of the "green" or molded abrasive article can be accomplished without having to resort to firing in special atmospheres, and which vitrifiable bond will have thermally-responsive expansion characteristics so related to those of the diamond abrasive that bonding and holding of the diamond particles in the final product will not be detrimentally affected by operational temperature changes which the grinding wheel undergoes and which vitrifiable bond will not detrimentally affect, during firing, the shape and volume of the molded article itself. Abrasive grains of a wide variety of natural and artificial substances or materials have heretofore been used or proposed to be used in the making of abrasive articles and they range not only from substances relatively soft to the hardest substances known, namely, diamond, but they also range or differ widely in their respective thermal coefficients of expansion, and hence they are not usually usable interchangeably with the same vitrified bond to produce safe or operative or efficient grinding wheels. For example, where one abrasive material would not produce crazing or like fracturing of the vitrified bond, another, in the same bond, would. Moreover, in a multiple or two-part grinding wheel, the thermal expansivity of the abrasive annulus as a whole would be materially altered by substituting in it abrasive grains of different thermal expansivity with respect to the thermal expansivity of the back or support which is not intended to be abrasive in action, with the result that undue strains or stresses could be created at the junction between the two parts. Another object of this invention is to provide a method of making a vitrified-bonded diamond grinding wheel by which maturing of the bond may be effected at temperatures below the oxidation temperature of the diamond particles without giving rise to detrimental or injurious differences in thermal expansivities.

Another object is to provide a vitrified bond of relatively soft composition, capable of strongly holding or bonding the diamond grains against the reactions from their cutting action, and capa-

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ble of substantially exhausting the cutting capabilities of the diamond grains and yet give way or itself wear away in a manner to achieve substantial uniformity of wheel wear and thus lessen the frequency of truing during the life of the grinding wheel. Another object is to provide a simpler and less expensive method of making vitrified-bonded diamond abrasive articles, and to provide a product resulting from such method that will have improved and desirable structural and functional characteristics. Other objects will be in part obvious or in part pointed out hereinafter.

The invention accordingly consists in the features of construction, combinations of elements, arrangements of parts, and in the several steps and relation and order of each of said steps to one or more of the others thereof, all as will be illustratively described herein, and the scope of the application of which will be indicated in the following claims.

I have found that oxidation of diamond particles, when included in a mix of suitable vitrifiable bond, takes place during firing in an atmosphere of air at temperatures above a value of 750° C.; when the maturing temperature of the bond is above that value, the effects of oxidation of the diamond particles become evident in various ways and to varying extents. For example, microscopic examination shows discoloration of the bond structure, apparently by by-products resulting from the oxidation or alteration of the diamond, and then again the effects of attack upon or reaction with the diamond sometimes appear in the form of bloating of the vitrified article, due probably to the evolution of gases resulting from the reaction with the carbon of the diamond.

In accordance with my invention I am enabled to avoid or materially diminish such undesirable effects as those above noted, without having to resort to expedients such as the incorporation in the mix of easily oxidized ingredients and/or the use of a non-oxidizing atmosphere and thus I am enabled greatly to simplify the production of vitrified-bonded diamond abrasive articles, and also to achieve certain other desirable advantages. According to my invention I have discovered that I can fire the molded mix of diamond particles and bond ingredients in an oxidizing atmosphere such as air, without material risk of oxidation of the diamond particles. This unexpected result I am enabled to achieve, in part, by first making a up a glass or glasses to have certain characteristics later set forth, and to such glass or glasses I then add other ingredients, including diamond abrasive particles for the

abrasive part, to make up a mix out of which the abrasive article is then molded and is then fired in the manner later described. According to my invention I provide a range of glasses to meet various requirements which glasses can be matured without recourse to such expedients as those above mentioned. A number of examples are hereinafter set forth.

As one illustration of a glass according to my invention, I make up a mix of silica and lead oxide, in suitable proportions and under conditions and treatment which are later set forth. This glass may be characterized as a lead silicate glass and it may contain alkali metal oxide. The ingredients of the mix, to which may be added other ingredients later mentioned by way of illustration, are thoroughly mixed together, heated to a relatively high temperature, preferably to about 1300° C. but at all events above the final maturing temperature of the article, under conditions later described, and held at such a temperature until the ingredients are melted together. Upon subsequent cooling the glassy product thus formed is crushed to particle sizes very small, on the order of 100 grit size or smaller. The resultant glass frit, depending upon what other ingredients are initially added to the mix, has a softening or maturing point at or below 750° C. and other characteristics suitable to functioning according to my invention. Whenever I refer to "softening" herein, I mean softening, sufficiently for good bonding. I am enabled, as is later described, to provide frits of suitable or desirable diamond bonding characteristics with predetermined softening temperatures; for example, I can provide a frit or glassy bond that may be matured during firing at a temperature on the order of 600° C. in an oxidizing atmosphere and this is particularly advantageous where the diamond grain is exceedingly fine and thus is more susceptible to oxidation under heat treatment. I also may provide a glass bond that may be matured at a somewhat higher temperature, for example, up to 750° C., and such a bond may be employed advantageously particularly for diamond in larger grit sizes having less tendency to be oxidized in an oxidizing atmosphere.

In making up the frits I prefer to avoid the existence or production of free lead in the glass; hence it is preferable that the lead oxides employed be devoid of free lead. For example, I may preliminarily treat the lead oxide as by heating it in an oxidizing atmosphere to insure the conversion of any free lead to lead oxide. Any of the lead oxides may be employed such as PbO although it is preferred to employ the more stable higher oxides such as Pb₃O₄.

In making up the mixes for the frits and heating the mix to bring the ingredients thereof into solution as above described, it is usually desirable to include in the mixes a suitable oxidizing agent to prevent the lead oxide ingredient from reducing to free lead, and desirably also the heating of the mix is for similar reasons carried on under oxidizing conditions as, for example, in an oxidizing atmosphere such as air. The inclusion of an oxidizing agent in the mixes for the frits may be effected by including alkali metal oxide in the form of a nitrate which under heat treatment of the mix furnishes oxygen to insure the oxidation of any free lead that might be initially present and also inhibits the reduction of the lead oxide to free lead.

Also, in making up the glass frit, it is desir-

able, during the heating cycle thereof, to achieve substantial release therefrom of gases or vapors so as substantially to avoid the production or evolution of gases during the subsequent firing of the vitrifiable bond and abrasive mix and an illustrative heating cycle may comprise heating the ingredients of which the frit is to be made to about 1300° C., in for example, a gas-fired furnace, under oxidizing conditions as above noted because of the lead ingredients, and maintaining that temperature until all of the batch is in solution and a clear melt is obtained, whereupon the temperature may be dropped to about 1100° C. and held at that temperature for a sufficient time to allow for the evolution and escape of the gases from the melt; the melt may then be quenched in water or be poured onto a suitable slab, allowed to cool, and then crushed and ground to the desired comminuted form such as 100 grit size or smaller.

Of the lead silicate glasses that may be made up according to my invention, for making up the frit, an illustrative composition may consist of lead oxide (either PbO or Pb₃O₄) about 70% by weight, silica about 27%, and alkali metal oxide such as sodium oxide or potassium oxide about 3%. The latter ingredient may, however, be omitted; it is included in any case in small proportion and primarily for the purpose of contributing its good fluxing qualities and thus better initiating the going into solution of the several ingredients when the mix is heated to make up the frit.

Using this frit, a two-zone grinding wheel may be made up with a center or back comprising a mix, by volume, of 50% of this frit and 35% of 100 grit size vitreous silica, molded under pressure and other conditions to give a volume porosity of about 15%, and having an abrasive annulus comprising by volume 50% of this frit, 18.6% of 100 grit size diamond, and 16.4% of vitreous silica of 100 grit size, molded under pressure and other conditions to give a porosity of about 15%; the two mixes for these parts are made up with the aid of a suitable plasticizer or binding agent appropriate to give the composite wheel structure when molded and pressed appropriate "green" strength so that it may upon removal from the mold be handled and trued or shaved as desired and then subjected to firing. The molding is preferably carried on under substantial pressure, on the order for example of from 20 to 25 tons per square inch. The molded "green" wheel may be dried in an oven, for example, at 90° C., overnight, and then may be fired at about 660° C. in air, bringing the temperature up to that value over a period of about four hours, maintaining it at the 660° C. for about two hours, and then allowing it to cool slowly. Upon comparative test, the wheel was exceptionally soft-acting when grinding cemented tungsten carbide.

In the firing of the above described wheel, no detrimental swelling or slumping took place, the firing temperature being approximately suited to the above described range of softening temperatures of the frit employed; moreover, no evidence of harm to the diamond grain was present even though the firing took place in an oxidizing atmosphere. Harmful gas evolution appeared to be absent and in this connection a factor lies very probably in the manner of preparation of the frit as above described, substantial expulsion of gases therefrom being adequately provided for. More-

over, I am also enabled to preclude harmful effects during firing by the employment of a temporary binder, in making up the "green" wheels, that insures the absence of residues capable of causing harmful effects during firing. I prefer to employ inorganic temporary binders such as magnesium bentonite which contains calcium carbonate and which I, therefore, preliminarily acidify with hydrochloric acid so as to get rid of the CO_2 which the carbonate would produce during heat treatment. The magnesium bentonite comes in the form of a powder. This is stirred with water and then acidified with hydrochloric acid. It should then be allowed to stand for a number of hours whereupon it forms a gel. Then the supernatant liquid is decanted.

A small quantity, on the order of 1% or so, of this inorganic gel I employ in making up the mix of frit, body material, and abrasive particles, for making up and molding the "green" wheel. Also, I may employ silica gel which is silicic acid (H_2SiO_3), or alumina gel which is $\text{Al}(\text{OH})_3$; these gels are also inorganic. Unlike organic binders heretofore employed, such as glutoline, glycerine, etc., etc., and which upon firing leave some residue, usually of carbon which can become oxidized into CO_2 during firing in an oxidizing atmosphere or in the presence of oxidizing ingredients or entrapped air in the "green" wheel and thus causing bloating, swelling or the like, the inorganic temporary binders that I may employ leave no residual material which could form a bloating gas and they are otherwise not harmfully reactable during firing. The molded "green" wheels, utilizing these temporary inorganic binders, are before firing preferably dried in an oven, for example, at 90°C . for ten hours or so, and these temporary binders break down into water which is expelled as a vapor during the drying and firing, and into substances which, during the firing, react with the glass of the bond composition or are dissolved in it. For example, the acidified magnesium bentonite gel breaks down into water, magnesium silicate, SiO_2 and some CaCl_2 which react with or are dissolved in the glass; the silica gel breaks up into water and silica and the alumina gel breaks up into water and Al_2O_3 . I may, however, also use plain water as a temporary plasticizing agent for molding the "green" wheel, the water being driven off during the oven drying that precedes the firing. The relative proportions of the silica and lead oxide may be widely varied, with or without additional ingredients of which the alkali metal oxide such as sodium oxide is an illustration; the silica may be present in percentages by weight from 25% to 60% and the lead oxide from 40% to 70%. Frits so made up have softening points broadly in the neighborhood of 600°C . as, for example, within a range from about 500°C . to 750°C . Ingredients like alumina may be included in small percentages in order to make the glass of the frit and hence of the ultimate bond less soft. The lead silicate frit glasses have thermal coefficients of expansion, being on the order of from 55×10^{-7} to 85×10^{-7} . When used as the glassy element of the vitrifiable bond, the molded or "green" abrasive article may be fired at temperatures appropriate to the softening temperature of the particular frit and hence within a general range of from 500°C . to 750°C ., and it will be noted that these maturing temperatures are below deleterious temperatures as above described.

I may raise the softening point as by adding a small percentage, on the order of 3% by weight,

of magnesium oxide (MgO); in that manner I may raise the softening point and substantially correspondingly elevate the maturing temperature by about 100°C ., but in any case the maturing temperature should be not above about 750°C .

With any of these lead silicate frit glasses I may compound the mixes for the abrasive part of the abrasive article and for the non-diamond-containing support or backing part thereof, by the addition of other ingredients appropriately coactive, particularly physically, with the glassy bond and, in the abrasive part, with the diamond abrasive particles, particularly in the light of relative thermal expansivities. For example, I prefer to employ, as a body material in either part, although it may be omitted from the diamond-containing part, a comminuted body material having a thermal coefficient of expansion as closely approximating that of diamond as possible. The expansion coefficient of diamond is about 11.8×10^{-7} and illustrative and suitable body materials to employ are vitreous silica or a highly refractory glass, preferably in grit sizes of 100 or smaller. These have the characteristic of softening points for bonding greatly in excess of the maturing temperature of the bond and they do not go into solution or otherwise react with the glass of the bond in a deleterious manner and instead are bonded by the latter as individual body material particles. In relation to the lead glasses of my invention, they respond to temperature changes without detrimentally affecting the glassy bond as by fracturing or crazing it or the like. They may be employed individually or in admixture.

As an example of a highly refractory glass suitable for use as above described, I may use a well known glass which has approximately the following formula: 80.6% SiO_2 , 13.0% B_2O_3 , 3.8% Na_2O , 0.4% K_2O , 2.2% Al_2O_3 . This glass does not soften at 750°C .

As above mentioned, the lead silicate glasses of the bond of my invention are relatively soft; nevertheless they strongly hold and bond the diamond particles and in this action they are aided by the included body materials such as vitreous silica, the particles of highly refractory glass, etc. Although the glass element of the bond structure is relatively soft, particularly as compared to vitrified bonds heretofore employed in diamond abrasive articles, the cutting action is freer than in the hard-bonded dense vitrified-bonded diamond abrasive articles heretofore employed. With its softness, the glass element of my bond structure appears to have also a quality of give or of relative yielding, which may be advantageous in avoiding breakage of wheels under high speed grinding conditions.

It will thus be seen that there has been provided in this invention a diamond abrasive article and method of producing the same in which the various objects above mentioned together with many thoroughly practical advantages are successfully achieved. The method of my invention will be seen to be simpler and less costly to carry on than methods heretofore employed and the diamond abrasive article will be seen to be highly advantageous in construction and action. Risk of harming the properties of the diamond grain which, of course, can be employed in various amounts or concentrations is practically absent.

As many possible embodiments may be made of the above invention and as many changes might be made in the embodiment above set forth, it is to be understood that all matter here-

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inbefore set forth is to be interpreted as illustrative and not in a limiting sense.

I claim:

1. A diamond abrasive article comprising diamond grains free from detrimental oxidation held in a substantially unswelled vitrified bond structure that comprises pre-fired lead silicate glass consisting essentially of by weight silica 25% to 60% and lead oxide 40% to 70% the article being vitrified at and its bond having a maturing temperature between 500° C. and 750° C. and said glass having been made by pre-firing at a temperature above that employed in maturing the article, and said article having a volume percentage of porosity not greater than 15 volume percent.

2. Method of making a vitrified bonded diamond abrasive article which comprises mixing materials containing silica and lead oxide, heating the mixture under oxidizing conditions to a temperature high enough to produce a glass, cooling the glass and crushing the glass to produce a powdered frit, the ingredients and the proportions of the ingredients of the mixture being such that the frit consists essentially of by weight silica 25% to 60% and lead oxide 40% to 70% and the frit has a softening point between 500° C. and 750° C., mixing diamonds with said frit, molding said diamonds and frit into a shaped article, and vitrifying said shaped article at a maturing temperature between said 500° C. and said 750° C.

3. Method according to claim 2 in which the

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materials that are heated to produce the glass and then crushed to produce the frit also contain alkali oxide but in proportion less than either of the silica and the lead oxide.

4. Method according to claim 2 in which the vitrifying is done under oxidizing conditions.

5. A diamond abrasive article according to claim 1 having a center comprising refractory glass bonded with the same unswelled vitrified bond that bonds the diamond grains.

6. Diamond abrasive article according to claim 1 also containing comminuted vitreous silica as an ingredient which becomes incorporated into the fired structure.

7. Diamond abrasive article according to claim 1 also containing comminuted highly refractory glass as an ingredient which becomes incorporated into the fired structure.

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