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CERAMIC ITEM DEFORMATION DURING FIRING: EFFECTS OF COMPOSITION AND MICROSTRUCTURE (REVIEW)

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The scope is considered for increasing the high-temperature stability to deformation in ceramic items as regards comprehensive improvement in characteristics corresponding to current economic trends.

Keywords: porcelain, deformation, buckling, high temperature, firing, sintering, viscosity, quartz, mullite.

It is becoming increasingly important to examine deformation during firing for molded semifinished products made of materials sintered at high temperatures and subsequently acquiring a vitrocrystalline nature, particularly on account of the tendency to reduce the firing times and temperatures by the use of suitable additives, and also in connection with tightening specifications for the size accuracy. Large ceramic products such as drainage pipes may be reduced in mass by reducing the wall thickness, which reduces the amount of raw material used and the energy consumption, but then there is an increase in the tendency of the semifinished product to pyroplastic deformation.

Researches have been done on a large scale on the creep in ceramics at high surface temperatures, but they have not been focused on the deformation in multicomponent systems such as porcelain during firing [1]. The variety of compositions and firing modes must determine the substantial differences in deformation [2]. It is not always possible to extend the phenomenology of deformation for materials whose elementary rheological properties differ substantially to other materials characterized by more complicated behavior. One approach is to examine porous vitrocrystalline material such as porcelain during firing, whose behavior is represented by a very complicated set of properties.

The task of giving a proper description of deformation splits up into several very complicated subtasks: determination of key parameters of the structure and composition, the changes in them during firing under load, the effects on the deformation, determination of the effects of firing conditions and shape of a component on processes, and this combines with the choice or creation of rheological models and software for calculating the deformation from the significant parameters.

The chemical and mineral compositions had the main effects on the deformation at high temperatures. Although there are many forms of porcelain, one can distinguish in their compositions the quartz component, whose grains are rarely greatly altered during firing, and which only partially dissolve in the liquid, together with the clay-mineral component and the fluxes, which form a liquid at a high temperature. One can control the properties of the porcelain, including the deformation behavior on firing, by adjusting the nature, grain size, and method of preparation for those components.

It is simplest to regulate the properties of a porcelain by varying the contents of low-activity crystalline phases: the oxides of silicon, aluminum, zirconium [3], and so on. One needs to consider not only the grain size but also the firing conditions, since the amounts of the crystalline phases may be reduced substantially by prolonged hold. There has been a study [4] of the reactions between porcelain components with model feldspar liquid, which has shown that in firing of duration 12 h with a hold of 1 h in the oven with electrical heaters the oxide of aluminum (0.83 μm) is almost insoluble in the feldspar liquid up to 1350°C, while the quartz (2.8 μm) dissolves to the extent of 10% of the initial material. On more prolonged heating in a gas-flame furnace, the amount of dissolving quartz was not more than 35%, or corundum 5%. The solubility of mullite formed in a mixture of metakaolinite with feldspar was high in both forms of firing. It has been found [5] that the pyroplastic deformation increases as the dissolution of the quartz in the glass phase increases and as the ratio of the length of the mullite crystals to their diameter decreases.

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There is considerable scope for regulating the properties of porcelain by selecting the grain sizes of the initial components. Finely divided mixtures as a rule provide higher physicomechanical and working properties [6, 7]. By increasing the dispersion of the initial components (quartz sand, feldspar, scrap porcelain, and so on) one can reduce the sintering temperature by 50 – 100°C [6 – 10]. The grain sizes of the quartz and other sparingly soluble components have the main effect on the sintering temperature of traditional porcelain, since the quartz grains act as solid inclusions and retard the sintering, which raises the maturation temperature of the porcelain [8, 9].

The sintering temperature can be reduced by replacing the quartz by sedimentary active silica [3]. The grain size of the feldspar has only a slight or inappreciable effect in reducing the temperature [10], whereas increasing the fineness of the quartz grains intensifies the dissolution. Due to the more extensive enrichment of the feldspar liquid with silica, the viscosity is increased and the sintering interval is extended [6, 7, 10]. Regulating the sintering interval for low-temperature porcelain has been considered [10] from the viewpoint not only of selecting the mean vein size (as has been established, the best grain size for quartz is 40 – 10 μm, while for feldspar grains it is less than 10 μm), but also with the best grain composition.

The viscosity tends to increase continuously on account of enrichment in silica by interaction with the liquid phase formed at elevated temperatures in increasing amounts. Consequently, there is relatively low sensitivity to composition changes at the stage of the sintering state.

Three-point bending tests on porcelain specimens for pyroplastic deformation have established [1] that the size of the particles of inert phase (low solubility) is the controlling factor in the liquid-phase sintering stage, but not in the stage of the sintering state. The use of quartz grains in the initial charge for the porcelain (15 wt.%) of larger size (16 μm instead of 1 μm) reduces the deformation rate during sintering. The quartz particles with diameter less than 10 μm after completion of the sintering are in contact one with another and form a rigid skeleton, which is not observed in the microstructure with initial grain size for the quartz of 1 μm. After the end of the hold at the maximum temperature, a specimen with an initial grain size for the quartz of 16 μm contained quartz relicts of diameter about 2 μm. Then in a porcelain with a high content of liquid phase, small quartz particles (<2 μm) cannot form a strong framework, and the amount of them is insufficient at 4 wt.% for the viscosity to be influenced. By analogy with quartz, the additional 3 wt.% of alumina with a large particle size (10 μm) reduced the deformation considerably.

Smaller grain sizes for the quartz and feldspar improve the homogeneity of the vitreous phase and produce a more homogeneous structure for the sintered material [11]. It has been shown [6, 7] that reducing the grain sizes of the quartz sand, feldspar, and porcelain scrap increases the amount of vitreous phase and increases the final shrinkage (reduces the porosity), and also reduces the expansion coefficient and smooths out the volume changes on the $\alpha \to \beta$ quartz polymorphic transition. An important point is that increasing the dispersion of quartz and feldspar grains increases the translucency and whiteness of the porcelain and thus makes it more beautiful [11].

However, increasing the dispersion of these components adversely affects the molding of the material [12], which is dependent primarily on the clay mineral component.

The molding method controls the general initial structure, and consequently the initial rheological parameters, which influence the structure development during firing. It has been found [13] that the method of preparing the specimens has a substantial effect on the apparent viscosity with readily fusible clays in the temperature range 950 – 1120°C. The viscosities of clay specimens prepared by the plastic method are 3.5 – 10 times higher in that temperature range than in the semidry forming method. Three-point bending tests have been performed on porcelain specimens made by slip casting, where it was observed [1] that elevated density in the blank is controlled by the amount of water in the slip and can substantially reduce the deformation during sintering, but has no effect after the end of shrinkage. It was considered that the high deformation of a low-density specimen is due to the increased mobility arising from the increased capillary forces in the large volume of cavities by comparison with a dense specimen.

Thermally activated firebrick with special grinding can be used to produce a slip with elevated solid concentration while retaining the casting characteristics and stabilizing the composition of few-component raw mixtures. For example, use has been made [14] of a slip with 17.5% water obtained by firing at 950 – 1000°C applied to a two-component mixture consisting of 50% kaolinite-hydromica clay with 50% quartz sand and subsequent rapid cooling and grinding by the technology used with highly concentrated ceramic bonding suspensions. The resulting castings had elevated density and the fired components had higher physicomechanical characteristics, with the shrinkage reduced by about 40% with water absorption of 0.5% as against 1% in comparison with traditional porcelain slip.

As a rule, there are many problems between production and use of items associated with inadequate homogeneity. A major factor in making a material with maximally homogeneous structure is to provide conditions in the initial stages of formation that exclude the formation of defects in all subsequent technological operations [15].

Adjacent blocks (regions) differing in texture may be the cause of cracking at the boundaries during drying or firing on account of anisotropy in the thermal expansion for volumes with differing orientation in the anisometric crystals. Also, in the case of drying for such a material, stresses may arise and persist for an uncertain time during firing associated with anisotropy in the air shrinkage. Clearly, structure anisotropy influences other properties of the ceramic. This must be borne in mind although ceramic material is often considered as isotropic at the macroscopic level on account of its polycrystalline structure.

There may be local differences in structure associated with the shape of the item. Measurements have been made [16] on the effects of various parameters on the deformation of porcelain items at each stage in production. It was considered that the choice of item form is not as important when the optimal formation conditions are met, which in practice is hardly attainable. For example, if shape memory is the main cause of deformation, the principal significance attaches to the texture.

The plastic properties of clays and their high capacity to produce texture may explain the effects of the shaping method on the macroscopic anisotropy of properties in the ceramic based on clays and the behavior during firing. Textures are formed when the shaping masses have clay components, which makes it very difficult to produce isotropic semifinished products [17], but the effects of structure anisotropy may be weakened during firing (faience, porcelain) when there is a considerable content of fluxes [18]. The converse phenomenon may be observed if there is an inadequate amount of liquid phase.

Texture effects have been examined [19] for specimens of traditional porcelain shaped by extrusion, particularly as regards the mechanical strength and Young's modulus as measured at room temperature. The cleavage surfaces of thin pseudohexagonal crystals of kaolinite have a preferred perpendicular orientation with respect to the radius of the cylindrical specimen, and the feldspar crystals are partially oriented, while the quartz crystals are randomly directed. The mechanical properties measured along the axes of the extrusion differ appreciably. The difference is about 4% for raw specimens, whereas it is about 20% for fired ones. The values of Young's modulus differ similarly. This is explained by the preferential anisotropic direction of the mullite crystal growth associated with the initial orientation of the kaolinite crystals, which has been confirmed by radiographic structural analysis. Then the shaping conditions for the semifinished product have a substantial but secondary effect on the properties.

There are many papers on the detailed physicochemical interpretation of porcelain firing, of which we may note a book [20], which emphasizes the role of the feldspars, which begin to melt at 1118°C (sodium feldspars) and 1150°C (potash ones). It is considered [11] that the contents of $Na_2O + K_2O$ not more than 4.2% in low-temperature porcelain do not cause an increase in deformation on firing at 900 – 1350°C, but the tendency to deformation varies with the K/Na ratio [21]. It has been shown [22] on two compositions for traditional porcelain differing in K/Na ratio that the necessary maturation (estimated from the parameters of shrinkage, density, water uptake, bending strength for the fired specimens) for porcelain rich in soda feldspar is attained at a temperature lower than that for a material rich in potash feldspar.

It is considered [23] that the best temperature for firing traditional porcelain is determined by the characteristics of the liquid phase. There is a considerable effect on the glass formation from the structure of the minerals in the feldspar raw material [11]. There are decisive effects on the degree of sintering and physicochemical properties of materials derived from readily fusible clays not only as regards the amount and viscosity of the vitreous phase but also the phase composition of the newly formed crystals (mullite), which are dependent on the sintering conditions: firing temperature and isothermal hold duration [24].

In the two-component Al_2O_3-SiO_2 system, mullite is formed at temperatures above 1400°C and with a molar ratio $Al_2O_3/SiO_2 \approx 1.5$; the region of solid solutions extends [25, 26] from the composition corresponding to mullite as $3Al_2O_3 \cdot 2SiO_2$ to the composition $2Al_2O_3 \cdot SiO_2$. In three-component systems (and with a larger number of components), containing K_2O, Na_2O, CaO, Fe_2O_3 (FeO) or TiO_2, the temperature for mullite formation is reduced because of the acceleration of reactions on account of the liquid phase, which appears at relatively low temperatures.

There are various views on the temperature range for mullite formation in porcelain compositions [20]. For example, it has been observed [4] that mullite is formed in a 1:1 mixture of metakaolin and feldspar already at 940°C, i.e., at a temperature somewhat less than that required for pure kaolinite (980°C). It has been reported [27] that metakaolin is transformed to a structure of spinel type and amorphous silica in the range 950 – 1000°C, while primary mullite is formed from the spinel above 1100°C.

Research on mullite formation in porcelain has been concerned in the main with increasing the strength of the porcelain by replacing the traditional clay components by more active ones and increasing the total content of Al_2O_3 as far as the complete exclusion of traditional components [2, 5, 25, 26, 28 – 30]. For example, adding alumina to the porcelain mass raises the strength of an insulator considerably [2]. In [30], as the Al_2O_3 source use was made of the cheaper bauxite. It has been suggested [25, 26] that instead of alumina and other oxide additives one should use a mixture of hydromica clay (50 – 60%) with $Al(OH)_3$, in which it has been shown that mullite is most rapidly formed at 1150 – 1200°C, which is 150 – 200°C lower than in the formation of mullite from oxides.

On the other hand, it has been shown [1] that 3.0 wt.% aluminum hydroxide added to traditional porcelain reduced the amount of mullite from 24.6 to 16.2 wt.%, presumably because the theoretical viscosity of the liquid phase was thereby raised by about a factor 3. Nevertheless, the deformation rate in three-point bending was substantially reduced, particularly at the sintering stage. Then the effect of increasing the resistance to deformation from raising the liquid-phase viscosity exceeded the negative effect of retarding the mullite formation.

It has been shown [31] that after sintering porcelain is in essence a three-component system: feldspar relicts (residues), kaolinite relicts, and partially reacted quartz particles. Each component consists of particles (primary mullite, secondary mullite, quartz or cristobalite), and has associated glass of a certain composition. The sizes of the relicts and the quartz particles vary from about 1 to 50 μm in accordance with the sizes of the initial particles. Electron microscopy has been applied [32] to the vitreous phase in drainage and electrotechnical porcelain, which showed prominent layering in the vitreous phase with the formation of small spherical zones and larger zones of elongated form, which had elevated alkali contents and were dispersed in the main vitreous phase. The lower viscosity of the dispersed phase favored the growth of secondary mullite needles. The secondary mullite was formed above 1200°C at the reaction boundaries between the kaolinite and feldspar relicts [27, 32].

Various researchers have suggested that a high length or high ratio of length to diameter in the secondary mullite needles may provide considerable stability to deformation. This does not agree with the view taken in [31]. There it was established that the volume proportion of mullite in the kaolinite relict should be 4 – 45 times larger than in the feldspar ones. If one considers the coarse grains with associated liquid as relicts, each of which contains only one particle, the relative proportion of quartz in them is approximately equal to the proportion of solid phase in the kaolinite relicts. Concentrated liquid suspensions containing macroparticles such as aqueous slips made of clays show that the viscosities of kaolinite and quartz relicts should be much higher than the viscosities of feldspar ones, since the concentration of mullite in the feldspar relicts is very low, and they are in fact readily deformed liquid matrices for the composition system. Moreover, on prolonged hold at the maximum firing temperature, the amount of mullite in the sintered material has a tendency to fall by dissolution [4], whereas primary mullite is much more stable and secondary [27].

Porcelain with a special composition may have a microstructure in which the role of the mullite needles in the deformation is more substantial. Measurements have been made [33] on the sintering in a reducing atmosphere as influencing the plastic deformation of porcelain specimens containing up to 60% Al_2O_3 at a pressure of 0.5 MPa (5 kgf/cm^2). The observed low deformation rate up to 1500°C is explained in terms of the mullite needles embedded in the vitreous phase along the corundum crystals. It has been suggested than when the crystallization center for needle mullite is provided by a particle stable in the liquid phase, with the mullite needles rigidly attached to it in the radial direction, the apparent diameter will be increased considerably and the effective concentration of the rigid phase becomes sufficient to increase the viscosity or even to form a three-dimensional framework. On the other hand, the role of the needle mullite crystals increases when one uses mineralizing additives, when the flow and volume of the liquid phase become comparatively large.

Optimizing the firing mode is always a problem. There are two groups of papers in this area that differ in their purposes: improving the properties of the material [34] or various economic parameters resolving problems in high-speed firing [35]). As a rule, the technological measures employed for this increase the buckling of the items, so the scope for them is restricted by the permissible deformation, and forecasting the deformation is rarely considered as a potential for improving the technology. Also, the optimal conditions determined for a material of one composition may be different for another. It has been found [36] that the heating rate (firing mode) has variable effects on the properties of ceramics made from various clays. Studies have been done [35] on the effects of the temperature gradient in the cross section of the furnace on the deformation of porcelain vessels on high-speed firing with maximum temperatures in the range 1150 – 1350°C and heating rates of 6 – 8°C/min. The maximum permissible temperature difference has been recognized as about 60°C.

When novel readily fusible components are included in the porcelain charge, there is a danger of excessive increase in the deformation of the sintered semifinished product on account of inadequate rate of crystallization in the latter stages of sintering in the absence of a strong framework and reduced viscosity in the liquid phase. Mineralizing additives are used to increase the general viscosity of the sintered material. For example, to reduce the sintering temperature and increase the general viscosity it has proved of value [34] to replace quartz and feldspar by glasses similar in composition to the vitreous phase in the porcelain but differing in their tendency to directional crystallization with the formation of a strong microcrystalline structure. Additives that initiate crystallization include CaF_2, MgF_2, and AlF_3. When only the feldspar is replaced, there is no substantial reduction in firing temperature. However, when quartz is replaced, the maximum firing temperature is reduced by 150 – 200°C and the mechanical strength is improved. The best composition is one in which 100% of the quartz and up to 75% of the feldspar are replaced by crystallized glass.

The addition of scrap glass containing lead [37] had a slight effect on the microstructure, while the use of a soda-calcium-silicate glass accelerated the formation of liquid phase and led to the crystallization of wollastonite, plagioclase, and sodium silicates, while reducing the contents of quartz in mullite and reducing the sintering temperature required to attain complete consolidation.

A porcelain that sinters below 1000°C can [38] be based on mixtures of kaolin and $CaCO_3$, where studies were made on the effects of borosilicate glass added as a mineralizing additive, as well as traditional components such as quartz and feldspar as regards the sintering characteristics. Borosilicate glass reduced the interval between the sintering temperature and the crystallization value, which reduced the thermal deformation, which previously had been impermissibly large. The addition of up to 25 wt.% quartz provided a dense porcelain body consisting of crystals of anorthite, wollastonite, helenite, and quartz, and it also reduced the shrinkage and thermal deformation.

Extensive research has been done on the sintering parameters and also the structure formation from mineralizing additives TiO_2, ZnO, and CaF_2 [39], or a mixture containing zinc oxide and magnesium carbonate [40]; other studies have been made on the production of mullite in alumosilicate systems in the presence of Fe_2O_3 (FeO) [25, 26]; on the effects of pyrite nodules on the sintering and high-temperature viscosity of a refractory clay [41]; and on features of the phase transformations on firing clay materials with elevated contents of iron oxides [42].

In [43], there is a detailed consideration of the effects from mineralizing additives on the structure and properties of oxide ceramics, porcelain, and glasses. It was found that in porcelain, those additives initiate the formation of mullite and the transformation of quartz to cristobalite and also produce structural stresses. Mineralizing additives that strengthen the porcelain include fluorides, boric acid, and phosphoric acid, together with their salts and anhydrides, as well as oxides of the alkaline-earth metals, and oxides of Mn, Fe, and Be. Boron phosphate has a particular place, since it leads to the rapid formation of cristobalite and mullite.

Impurities and the gas composition have important effects on the physicochemical processes in firing ceramic materials, in addition to those of deliberately added substances. Studies on these are not numerous and are of particular local character. A reducing medium favors silicate formation at lower temperatures. Various impurities may substantially reduce the viscosities of clays for certain gas compositions [13], which makes it necessary to monitor the constancy of the gas medium in researching the thermomechanical properties of the material.

The published data indicate the following general concepts.

The structure of porcelain at the end of high-temperature hold may be considered as a composite composed of rigid kaolinite relicts, relicts of quartz crystals, and crystals of secondary mullite distributed in a matrix of feldspar liquid. The viscosity of the composite is controlled by the concentration of the residual quartz grains and two-phase aggregates containing kaolinite relicts, not by the concentration of crystals of secondary mullite. In general, the roles of mullite and other components are determined by the microstructure: morphology and concentration of the solid particles, the relative positions of them, the microstructures, the viscosity of the liquid phase, and so on. Raising the density of the blank increases the resistance to deformation on firing. Researching the rheology of an anisotropic medium is a complicated practical and theoretical task, but it provides good potential for controlling the deformation and other properties.

The basic way of optimizing the composition is dependent on the number of sintering additives in the basic material. In any case, one needs a fraction of relatively coarse quartz particles or other inert filler to provide a rigid framework at the stage of liquid-phase sintering. The effective sintering stress (shrinkage) should be maximal throughout that stage. This can be provided by a high surface tension for the liquid phase, good wettability of the grains, optimal amount of a liquid, and proper variation during the pore filling.

If the material on firing produces a large amount of liquid phase on low viscosity, one should raise the total viscosity by accelerating the growth of new crystals with controlled orientation. These form rigid branched substructures that substantially increase the effective solid-phase concentration, which favors increasing the total viscosity or produces a strong framework.

In the case of porcelain with a low content of eutectics, it is desirable also to increase the deformation stability by raising the viscosity of the liquid phase. The sintering is best intensified by increasing the amount of liquid phase, not by reducing its viscosity. For example, instead of increasing the content of sintering additives that form eutectics with low viscosity one should prefer to add a finely divided silica fraction.

The length of the high-temperature firing stage should be determined by the required final porosity as a maximally limiting factor in the rate of porcelain maturation. All necessary high-temperature structure transformations should be completed in that time. This can be provided in various ways, including by optimizing the composition and the firing mode. For a porcelain with a high content of eutectics, it is usually impermissible to exceed a certain high-temperature stage, since that may lead to falling viscosity because the crystals dissolve.

Usually, the deformation (for example, in creep) is considered as a function of temperature, time, stress, and structure. However, the deformation during firing is a function of many additional factors such as the chemical, mineral, and grain-size compositions of the initial components, the density of the blank, the heating rate, the reactions between components, and so on.

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