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Ceramic Tile Strengthening By Sintering Process

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Abstrak

Penambahan kekuatan pada keramik dengan cara sintering memainkan peranan yang penting didalam pembuatan bahan keramik lantai guna memproduksi produk keramik yang dapat diandalkan pada teknik sipil. Hal ini tidak lain berdasar pada suatu kenyataan bahwa transpor elemen material didalam strukturmikro selama proses sintering diakibatkan oleh proses difusi permukaan dan difusi didalam kisi kristal. Proses difusi inilah yang menentukan penambahan kekuatan pada bahan keramik pada industri konstruksi sipil. Didalam penelitian ini struktur dari elemen material dalam bentuk bubuk sebagai bahan dasar dan juga bahan aditif berupa slag dianalisa dengan teknik difraksi sinar-x. Keramik bubuk dan aditif slag dicampur dengan perbandingan 5-50% untuk mempelajari efek dari proses sintering. Penambahan kekuatan pada keramik diamati berdasarkan beberapa indikator seperti penyusutan volume, porositas, uji kekerasan, dan kekasaran permukaan. Setelah bahan disintering sampai temperatur 1000^0 C bahan dianalisa dengan bantuan teknik difraksi sinar-x untuk mengetahui struktur kristal serta transformasi struktur mikro. Hasil analisa difraksi sinar-x menunjukkan bahan dasar keramik terdiri dari silika dan albit, sedangkan bahan aditif terdiri dari $NiAs_2$. Keramik yang mempunyai porositas terrendah, kekerasan tertinggi, penyusutan volume tinggi dan kekasaran permukaan yang baik didapat dengan penambahan bahan aditif sebesar 5%. Dengan demikian dapat disimpulkan bahwa mekanisme penguatan pada keramik terjadi karena terbentuknya albit sebagai hasil penambahan aditif $NiAs_2$ sebesar 5%.

Abstract

Ceramic strengthening and sintering process plays important role in the fabrication of ceramic tile as well as to produce reliable ceramic products for civil engineering application. This stem from the fact that microstructures elemental materials transport during sintering process by surface and lattice diffusion governed the strengthening mechanism. In these studies, structure of elemental materials in the form of powder ceramic as well as its additive (slag) has been analysed by x-ray diffraction technique. Powder ceramic, and slag additive is mixed all together with a ratio of 5-50% in order to determine the result of sintering process. Strengthening was observed and analysed base on several indicators such as; volume shrinkage, porosity, hardness, and surface roughness. After sintering at temperature of 1000 ^{0}C, the product is analysed with x-ray diffraction technique to determine phase change as well as microstructures transformation.

X-ray diffraction study indicated that the base materials consist of silica and albit and the additive materials are $NiAs_2$. The ceramic material of lowest porosity, highest hardness, high shrinkage and good surface roughness were obtained with 5% additive material. It is concluded that the strengthening mechanism is governed by the formation of albit, as a result of additive consist of 5% $NiAs_2$.

Introduction

Lately, housing ceramic products have been developed for a wider application for both consumer goods as well as for civil industry applications. The main reason for this is due to the development of analytical instrumentation's capable to control the result of sintering process in more systematically fashion both structurally as well as chemically, namely by x-ray fluorescence (XRF) and x-ray diffraction (XRD). In many applications, its insulator properties are more significant than mechanical properties. Increasing demand of new material to resist harsh temperature regime such as gas turbine application require not only insulator properties but also mechanical properties. Therefore it is not surprising that in recent time both of its properties has been considered equally. In general, ceramic has several distinctive characteristics such as resistance to compressive deformation, high hardness, and resistance to wear in high temperature environment [1]. Nonetheless, the only draw back of ceramic is its susceptibility to brittle fracture. To reduce its brittleness requires some modification in the composition, due to the fact that fracture initiation is related to atomic bond breaking. By changing its composition the chemical bonding will improve to such extent that it is able to accommodate thermal shock at severe environment particularly at high temperature. Reducing its grain boundaries structural unit such as; by solid solution strengthening, precipitation strengthening and process treatment can increase ceramic strength. The method used in this investigation is through process fabrication procedural such as: increase densification by pressing dry ceramic powder.

Materials and Method

Materials were prepared base on ball clay obtained from ceramic tile manufacture. The ball clay is a standard material for ceramic tile base material. Subsequently the base materials were mixed with slag obtained from by product of nickel mate refining process with vacuum arc furnace. The additive contained As, Ni, Cr and V with the remaining of calcium referred here as additive with mixing ratio of 0%, 5%, 10%, 15%, 20%, 30% 40% and 50% by weight to the base materials (ball clay). The material was drying and mixed mechanically for 1 hour and then is placed in the mould (coin shape) with 2 inches diameter. Subsequently it is pressed to increase its densification at 1.5 ton/cm. Thereafter is heated by heating to a temperature of 1000^0 C for three hours. X-ray fluorescence and X-ray diffraction examined the volume shrinkage, porosity, surfaces and bulk hardness as well as its structural change.

Result

The result of this experiment is tabulated in the Table 1 and Table 2. Table 1, show the qualitative analysis result of elements obtain by x-ray Fluoreseence technique with the parameters. A qualitative Analysis gave more information of the elements

formation during sintering process whereby surface diffusion in the main transport vehicle. Table 2, show the x-ray diffraction analysis of the materials (base, additive and mixed materials).

Table 1. Qualitative X-ray Fluorescence Analysis of the materials.

Material	Target	Voltage (kV)	Current (mA)	Full Scale (kCPS)	Angle (2 0)	Elemental composition
Base	PET	35	30	5.0	10-80	Fe, Mn, Ti, Ca, K, S, P, Si, Al, Mg,Na
	TAP	35	30	16.0	10-80	
Additive	PET	35	30	10.0	10-80	Fe, Mn, Ti, Ca, K, S, Ni, Cr, V
	TAP	35	30	15.0	10-80	

Table 2. X-ray Diffraction Analysis of the materials

Parameter	Instrument setting	Material	Phase Identification	Structure
Type of sample	Ceramic Powder	Base	SiO_2	Hexagonal
Applied voltage	40 kV		Albit $NaAlSi_3O_8$	Triclinic
Current	20 mA			
Temperature	Ambient	Additive	$NiAs_2$	Orthorombic
Wave length	1.5405 A			
Target	Cu K			
Detector	Scintillation	Mixed	SiO_2, $NaAlSi_3O_8$, impurity	
Range	2kCPS			
Filter	Ni (nickel)			

Mechanical properties and volumetric shrinkage measurements were conducted with several different compositions by mixing base material with additive ranging from 0-50% of additive material. Volume shrinkage, porosity and surface hardness were measured. Surface roughness and surface hardness were measured by using surface hardness tester Type M with Vickers indentor. The result is tabulated in the Table 3.

Table 3. Volume shrinkage, Porosity, Surface Roughness and Hardness of ceramic base with 0 – 50 % of additive material.

Base	Additive	Shrinkage (%)	Porosity (%)	Roughness (um)	Hardness (grf/um)
100	0	3.7324	23.4508	2.00	307.89
95	5	7.5619	16.4849	1.50	563.77
90	10	6.8301	16.8488	1.75	492.20
85	15	6.7958	17.4877	2.00	447.62
80	20	6.1372	18.6186	2.25	380.81
70	30	5.2361	19.8661	2.50	360.36
60	40	3.8994	21.1062	2.75	329.98
50	50	3.2152	21.9409	3.00	321.40

The pictures of surface microstructures observation were taken with Olympus inverted metallurgical microscope for material specimen with 5% ratio of additive material to the base material (Figure 1).

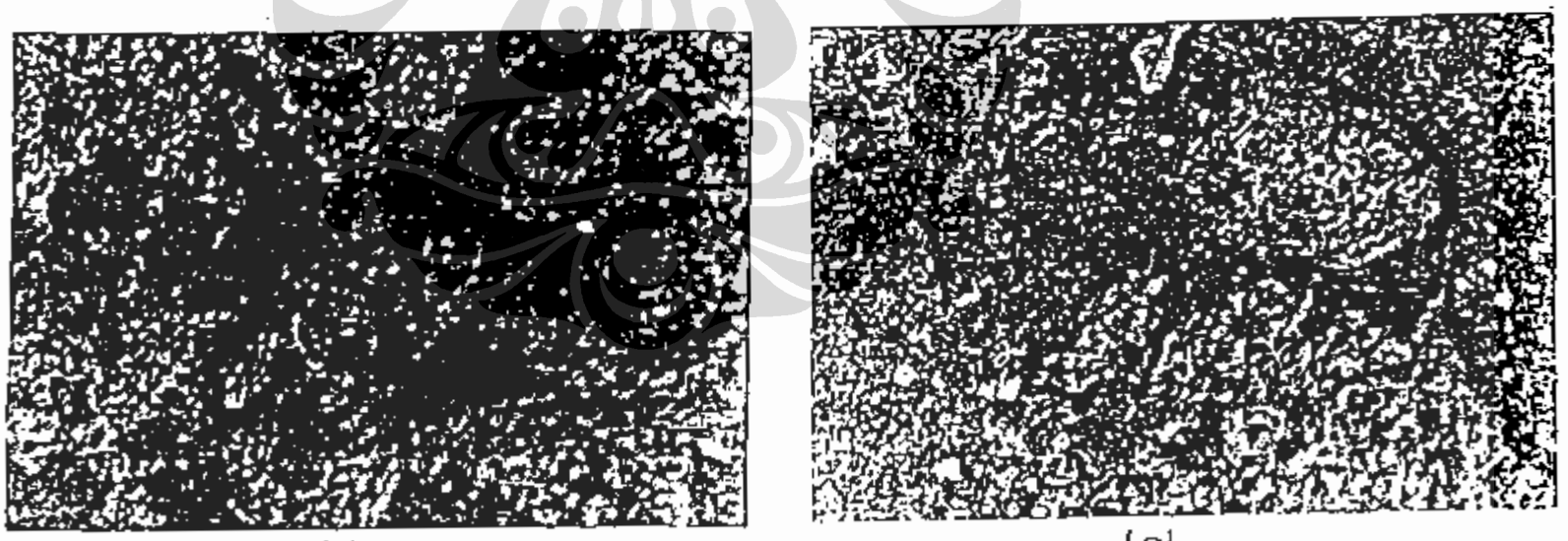

Figure 1. Micrograph of ceramic with 5% additive material. x 500.

Discussion

Ceramic consisted of inorganic and non-metallic materials, bound atomically by both covalent and ionic bonds. Ceramic can existed as a simple compound, and up to several phase of a compounds which form a complex structure. Pressing and subsequently firing without having to go to a drying process can do as the procedure of fabricating ceramic. The process is depicted in Figure 2. During firing process ceramic base material under go a physical change both macro - scopically as well as microscopically. This translated into a change in grain size, pore

shape and pore size, which in turn influence its physical form as well as mechanical properties. From Table 3, it can be seen that with 5% additive of NiAs to the base materials give the highest hardness, lowest porosity and yet high shrinkage. The result of the firing process cause a change in weight and volume, as well as reduction in atomic surface area, thus increases in surface contact as a result surface diffusion (material transport) and finally its grain size.

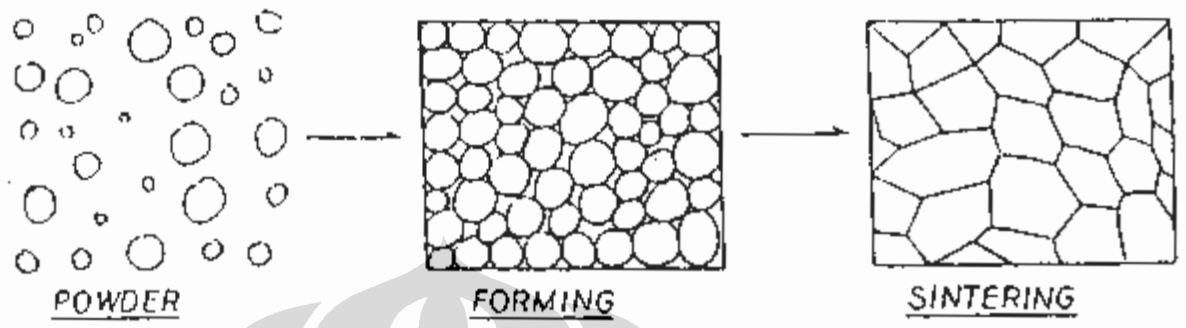

Figure 2. The procedure of fabricating ceramic

Theoretically, the densification process of ceramic powder prior to firing can be described both through vapour phase process as well as in the form of solid condition, which is depicted in Figures 3.

Figure 3. The densification process of ceramic powder

During sintering process, the vapour pressure generally is higher on the particle surface with positive radii of curvature than on the flat surface[2]. During sintering process, the boundary between two particles formed and generally is known as necking with negative radius of curvature. On the particle surface with negative radii of curvature the vapour pressure is lower. The difference in vapour pressure cause material transport into the neck area of two particles. The rate of bonding at the neck of two particles is governed by the rate of material transport into that area, which in turn determine to volume of material transport[3]. The material transport to the neck area occur by lattice diffusion as well as surface diffusion, and the rate of transport is determined by the difference in vapour pressure between two radii (positive and negative) of curvature. During diffusion process the distance

between the centre point of two particles does not change but remain the same, and also does not shrink the particle. Nevertheless, the volume of material transport to the neck between two particles has a significant influence to the pore shape. Material transport to the neck between two particles occurs by lattice diffusion. The rate of material transport is equal to the rate of material release to the neck area, and this in return would determine the rate of sintering at the vapour phase. The volume change during the sintering process can be written as follows:

$$V/V_0 \quad 3\ 1/l_0 - 3\ \{20\ a^3\ D^*/2^{0.5}\ k\ T\}^{0.4}\ r^{-0.5}\ t^{2.5}$$

The formulation indicated the increasing bond between particles, in which the bond is increase by one fifth of a time. X-ray diffraction analysis were taken in order to study its structural behaviour for both base and additive as a standard procedural[4]. X-ray diffraction study indicated that the base materials consist of silica (hexagonal structure) and albit (triclinic). The additive materials are NiAs with orthorombic structure and the strengthening factor of increases in compressive strength.

The photomicrograph result as well as several measurements conducted in this investigations that the best result in term of low porosity, high hardness, low percentage of shrinkage and good surface roughness were obtained with 5% additive material. This indicated that by properly adjusting composition, sintering time and temperature, excellent ceramic properties can be obtained through good understanding of the process and mechanism that govern the strengthening mechanism. The process carried out in this investigation as well as using some additive material, it can be used for heavy-duty application.

Conclusion

X-ray Diffraction result indicated that the strengthening mechanism is governed by the formation of $NaAlSi_3O_8$. (albit), as a result of additive consist of $NiAs_2$ phase. By adding 5% additive material, excellent properties of ceramic tile can be obtained for civil engineering application.

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