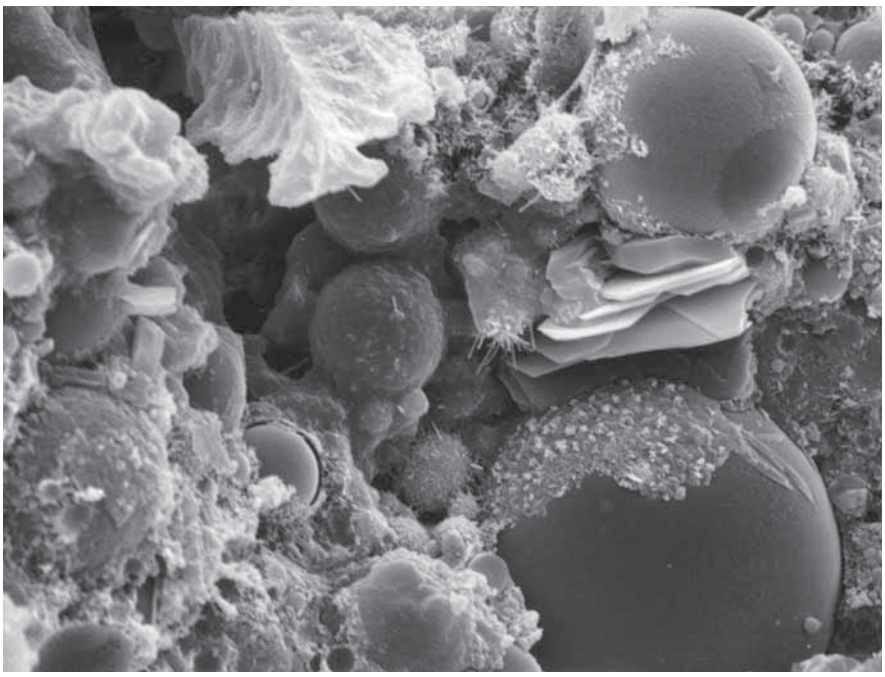
Proceedings of the International RILEM Conference

Materials, Systems and Structures in Civil Engineering 2016
Segment on

Concrete with Supplementary Cementitious Materials



Edited by
Ole M. Jensen, Konstantin Kovler and Nele De Belie

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PRO 113

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Photo 1st cover page: Fly ash particles embedded in different reaction products. Width of photo approximately 650 µm. Credit: Keren Binyamin & Konstantin Kovler (Technion)

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Contents

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SINTERING OF CERAMICS BASED ON MECHANICALY ACTIVATED FLY ASH

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Abstract
This paper presents the results for the influence of the mechanical activation on the properties of the sintered fly ash compacts. By varying the time of mechanical activation (short time of 10, 20 and 30 min) and temperature of sintering: 1050, 1100 and 1130 °C/60 min a spectrum of properties was obtained, but as an optimal defined was the mechanical activation of 20 min prior to the sintering at 1130^0C.

1. Introduction

Solid waste (fly ash and bottom ash) generated from the thermal power plant during the combustion of coal to produce electricity presents the global problem from the environmental point of view. Part of the generated fly ash is successfully used in the cements production [1], but also the possibilities to incorporate fly ash into ceramics matrices like in tiles [2] and bricks [3], in the production of mullite [4] as well as in geopolymers [5] are widely reported. Fly ash presents valuable low cost material due to its chemical composition consisting mainly of SiO_2, Al_2O_3, CaO, Fe_2O_3. Physical properties of fly ash depend on the burning conditions and the type of coal. The shape of the fly ash particles are mostly spherical with specific surface area from 250 to 600m^2/kg. As far as it is very fine material it is usually used as raw material with no pre-treatment [6, 7]. By increasing the geometrical factor of activity – specific surface area and reducing the particle size distribution the consolidation of the powder is favored, usually at lower temperatures.

This paper presents the results obtained from the investigation realized by pre-treating the fly ash i.e. thermal treatment (600^0C) and mechanical activation for short period of time (10, 20 and 30 min). Also, the influence of the pretreatment on the final properties and the microstructure is discussed.

2. Materials and methods

Fly ash used in this investigation was obtained from the thermal power plant REK Bitola, Republic of Macedonia, derived from lignite combustion. X-Ray Fluorescence (model ARL 990XP) was used for determination of the chemical composition of fly ash. The loss of ignition (LOI) was determined by calcination of pre-dried samples at 900^0C/2h. Residual coal contained in the ashes was determined from the mass loss after 2 hours at 600^0C. Particle size distribution of the fly ash was determined by sieving analyses (Retsch AS200) and the specific gravity was calculated using the pycnometer method.

The morphology of the fly ashes during the investigation was followed by scanning electron microscopy (Leica S 440I) coupled with EDS, which was used for determination of chemical composition of particular fly ash particles.

The thermal properties of the pressureless prepared fly ashes compacts were determined using a heating microscope (Leitz Wetzlar) in the temperature interval of RT-1400^{o}C, in air atmosphere with a heating rate of 10 ^{o}C/min. Dilatometry (NETZSCH 402E) was used to follow the shrinkage of the pressed fly ash samples during sintering from RT to 1130^0C.

In order to increase the structural and geometrical activity of the fly ash, mechanical activation was applied in vibro mill. Mechanical activation was applied for a short time (period of 10, 20 and 30 min) in order to follow the obtained properties on the sintered fly ash compacts. The fly ash samples in this investigation were assigned as: TFA for thermally treated fly ash at 600^0C, and TFA-MA10, TFA-MA20 and TFA-MA30 for the fly ash samples thermally treated and mechanically activated for 10, 20 and 30 min prior to the sintering.

Fly ashes were consolidated by uniaxial pressing (P = 45 MPa) using 8% water as a binder. The compacted green samples were dried at 105^0C for 10 hours prior to the sintering. The sintering of the TFA samples was realized at 1050, 1100, 1150 and 1200^0C, but for the mechanically activated fly ashes (TFA-MA10, TFA-MA20 and TFA-MA30), the sintering was realized at three different temperatures: 1050, 1100 and 1130^0C using chamber furnace. The applying heating rate was 10^0C/min. The isothermal treatment at final temperature was 60 min. The cooling to RT was not controlled.

Water displacement method according to EN-993 was used for determination the bulk density. Porosity was calculated from the relative density.

The bending strength and E-modulus of the sintered fly ash compacts were determined with the 3-point bending strength tester (Netzsch 401/3) with 30mm span and 0.5mm/min loading rate. Instron testing machine (model 1126) with a crosshead speed of 0.5 mm/min was used for the compressive strength test. Three samples were used for determination of the mechanical properties and the average values were reported as the result.

3. Results and discussion

The chemical composition of the investigated fly ash is presented in the Table 1. According to the CaO which is 11.49wt.% this fly ash can be classified as C class fly ash according to the ASTM C618 definitions. The free CaO was not detected. Residual (unburned) coal contained in the fly ash was 2.75 wt.% .

Table 1: Chemical composition of fly ash

Oxide	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	LOI
wt.%	54.74	20.29	6.76	11.49	2.46	1.57	0.93	0.93	3.12

It is evident from the chemical composition presented in Table 1 that silica (SiO_2) and alumina (Al_2O_3) are the main oxides, but besides them, fly ash has a significant amount of calcium oxide (CaO) and iron oxide (as ferric). The integral content of SiO_2 comes from SiO_2 - quartz, $CaAl_2Si_2O_8$ – anorthite and $NaAlSi_3O_8$ – albite and according to the XRD the presence of Fe_2O_3 - hematite, $CaSO_4$ - anhydrite and an amorphous phase was also determined [8].

Fly ash belongs to the relatively fine materials and according to the granulometric composition, Table 2, almost 50% of fly ash particles are less than 63μm. The specific gravity of the fly ash is 2.17g/cm^3.

Table 2: Granulometric composition of as received fly ash

Diameter, (mm)	Fly ash, (wt.%)
+1.0	0.6
-1.0+0.5	1.5
-0.5+0.25	7.6
-0.25+0.125	18.0
-0.125+0.063	25.2
-0.063	47.1
Σ	100

The morphology of fly ash is presented in Figure 1. Figures 2-4 and Table 3 present the morphology and the chemical composition (microprobe analysis) of the Fe_2O_3, needle SiO_2 and amorphous silica (diatomite). Typical fly ash cenospheres are presented in Figure 3 and the rough surface particle presents hematite.

In the present investigation, in order to reduce the sintering temperature of fly ash, mechanical activation during short period of time (10, 20 and 30 min) was performed. Morphology of the

TFA particles and after mechanical activation (TFA-MA10, TFA-MA20 and TFA-MA30) are presented in Figures 5-8. It is evident that by increasing the time of mechanical activation the particles reduced their dimension. After 10 min of milling, part of the particles remain their start dimensions, but the biggest part of the particles changed their dimension and morphology. The dimension of the particles, TFA-MA10, varied from 2 to 50μm, but for the TFA-MA20 and TFA-MA30 the particles sized distribution varied from 1 to 30 μm and 1 to 20μm, respectively.

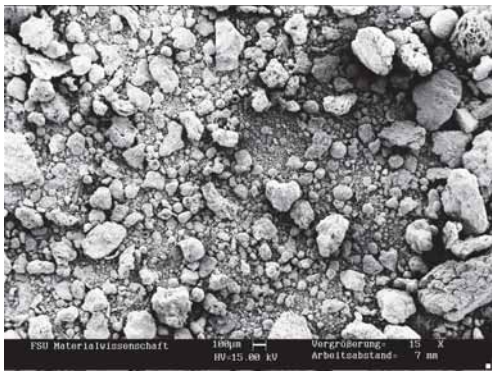

Figure 1. Morphology of fly ash (bar 1μm)

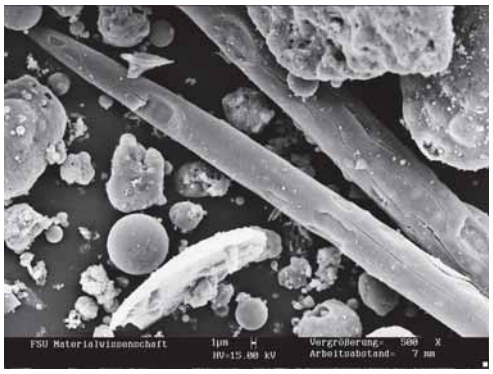

Figure 2. Morphology of needle SiO_2 (bar 1μm)

Figure 3. Morphology of Fe_2O_3 (bar 1μm)

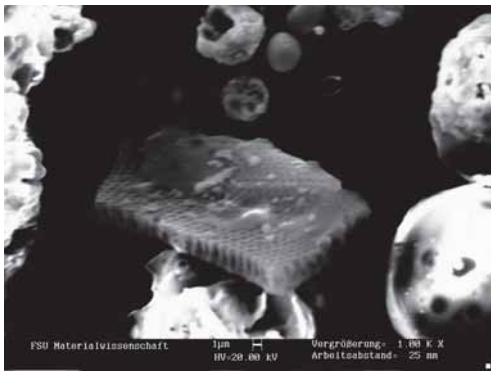

Figure 4. Morphology of diatomite (bar 1μm)

The thermal characteristics of pressure less prepartd fly ash samples before (TFA) and after mechanical activation (TFA-MA10, TFA-MA20 and TFA-MA30) are presented in Table 4.

Table 3: Chemical composition of the particular fly ash particles – needle quartz, hematite and diatomite

Oxide	Needle quartz, (wt.%)	Hematite sphere, (wt.%)	Diatomite, (wt.%)
SiO_2	98.12	1.75	95.48
Al_2O_3	0.56	0.13	1.62
Fe_2O_3	0.35	97.07	-
CaO	0.22	0.35	0.37
MgO	0.18	0.13	0.01
K_2O	0.06	0.11	0.20
Na_2O	0.33	0.07	0.70
SO_3	0.09	0.02	0.53
TiO_2	0.05	0.15	0.04
MnO	0.01	0.05	0.02
Cr_2O_3	0.04	0.08	0.50

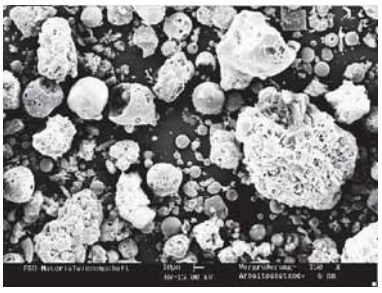

Figure 5. Morphology of TFA (bar 10μm)

Figure 6. Morphology of TFA-MA10 (bar 10μm)

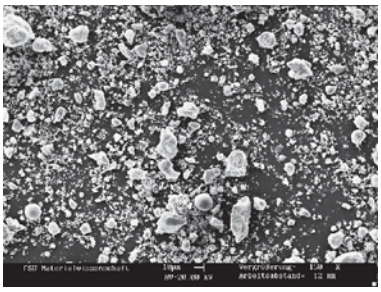

Figure 7. Morphology of TFA-MA20 (bar 10μm)

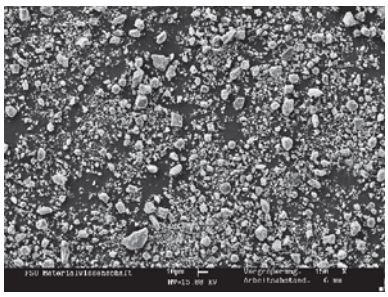

Figure 8. Morphology of TFA-MA30 (bar 10μm)

Table 4: Thermal characteristics of fly ash before (TFA) and after different time of mechanical activation (TFA-MA10, TFA-MA20 and TFA-MA30)

Type of fly ash	Significant shrinkage, ^{0}C	Softening temperature, ^{0}C	Melting temperature, ^{0}C
TFA	1220±10	1260±10	1360±10
TFA-MA10	1160±10	1220±10	1350±10
TFA-MA20	1150±10	1210±10	1340±10
TFA-MA30	1145±10	1215±10	1345±10

The region of sintering of fly ash before mechanical activation, TFA, is 1220 - 1260±10^{0}C, but it is reduced with the increase of the time of mechanical activation. The sintering regions for the mechanicaly activated fly ashes are: 1160 - 1220 ±10^{0}C for TFA-MA10, 1150 - 1210±10^{0}C for TFA-MA20 and 1145 - 1215±10 for TFA-MA30. The particle size of fly ash decreased during the mechanical activation and this results in the decrease of the melting temperature from 1360±10^{0}C for the starting fly ash (TFA) to 1345±10^{0}C for fly ash mechanicaly activated for 30 min (TFA-MA30).

After the consolidation of the fly ash powders by pressing, the shrinkage of the consolidated samples during sintering was followed by dilatometry. The dependances of the shrinkage with temperature in the polythermal part of sintering for the fly ash before (TFA) and after mechanical activation (TFA-MA10, TFA-MA20 and TFA-MA30) are presented in Figure 9. The shrinkage of TFA started at 400^{0}C and countinues slightly up to 1100^{0}C, reaching the total shrinkage of 2.61mm at 1130^{0}C. The shrinkage for mechanically activated fly ash samples (TFA-MA10, TFA-MA20 and TFA-MA30) starts at a temperature 730^{0}C and up to the temperature of 1050^{0}C it showed slight shrinkage followed by rapid shrinkage up to the temperature of 1130^{0}C. Mechanically activated TFA-MA20 sample showed maximal shrinkage of 4.72mm.

Sintered fly ash compacts were characterized from physical (density and porosity) and mechanical (bending strength, E-modulus and compressive strength) aspect. The properties of the sintered fly ash compacts are presented in Table 5.

Generally, by increasing the sintering temperature and the time of mechanical activation the density and mechanical properties increased, but the porosity decreased for the all sintered fly ash samples (TFA, TFA-MA10 TFA-MA20 and TFA-MA30).

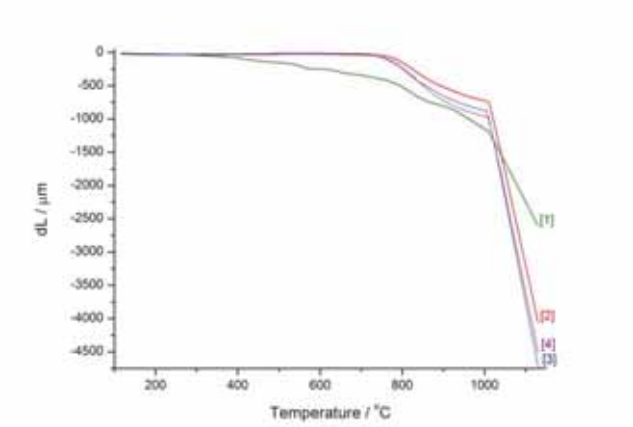

Figure 9. Shrinkage /temperature dependence in polythermal part of sintering for:
TFA, curve [1]; TFA-MA10, curve [2]; TFA-MA20, curve [3] and TFA-MA30, curve [4].

Table 5: Physical and mechanical properties of sintered TFA, TFA-MA10 TFA-MA20 and TFA-MA30 samples

Sample	T, ^{0}C	Density, g/cm^3	Porosity, %	E-modulus, GPa	Bending strength, MPa	Compressive strength, GPa
TFA	1050	1.25	47.01	2.17	1.71	2.49
	1100	1.29	46.89	1.88	2.67	2.55
	1150	1.46	44.07	6.73	7.27	8.32
	1200	2.01	1.66	21.34	35.47	32.30
TFA-MA10	1050	1.60	40.69	6.54	11.44	19.47
	1100	1.62	38.98	8.16	17.32	27.84
	1130	2.27	15.37	41.17	74.30	67.67
TFA-MA20	1050	1.61	39.98	7.13	11.95	19.65
	1100	1.63	37.64	9.94	18.51	31.53
	1130	2.34	11.46	48.04	79.90	78.48
TFA-MA30	1050	1.62	39.14	7.26	12.42	22.91
	1100	1.65	37.12	11.73	21.75	35.65
	1130	2.45	9.89	50.65	83.58	80.54

TFA compacts have low values for mechanical properties (up to 8.32 GPa compresive strenght for the sample sintered at 1150^{0}C) except the compacts sintered at 1200^{0}C. The values for mechanical properties are almost 3 to 4 times higher as the sintering temperature

increased from 1150 to 1200^{0}C. The comparable microstructures for the sintered TFA compacts at 1150 and 1200^{0}C are presented in Figures 10 and 11. The effect of the different degree of sintering is evident. At temperature of 1150^{0}C, part of the fly ash particles lost their individuality and the appearance of the liquid phase among the spherical fly ash particles is evident. Liquid phase sintering is favoured at 1200^{0}C.

The presented results for the mechanically activated fly ashes (TFA-MA10, TFA-MA20 and TFA-MA30), Table 5, showed that the properties increased significantly after 10 min of mechanical activation (in comparison to TFA) and there are no significant changes of the properties for 20 and 30 min of mechanical activation (TFA-MA20 and TFA-MA30). The values of density, porosity and mechanical properties have no significant differences for all fly ash compacts sintered at 1050 and 1100^{0}C, but the rapid changes of the values occurs for the fly ash compacts sintered at 1130^{0}C. The microstructures of the fly ash compacts (TFA-MA10, TFA-MA20 and TFA-MA30) sintered at maximal temperature of 1130^{0}C are presented in Figures 12 - 14.

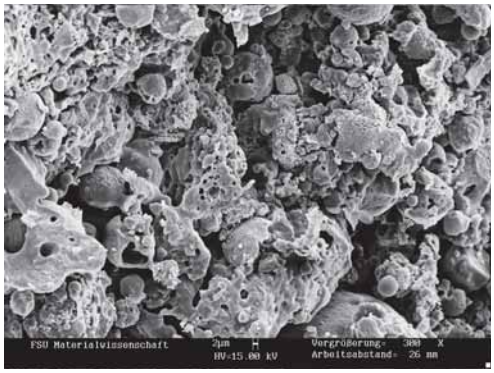

Figure 10. Microstructure of TFA compacts sintered at 1150^{0}C (bar: 2µm)

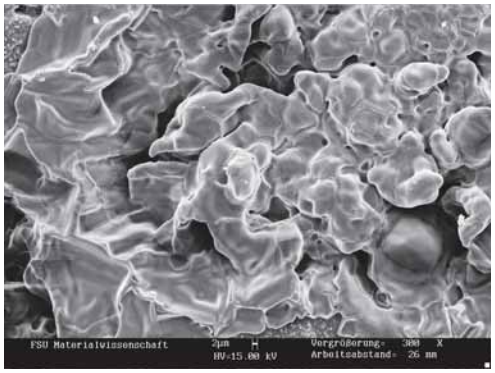

Figure 11. Microstructure of TFA sintered at 1200^{0}C (bar: 2µm)

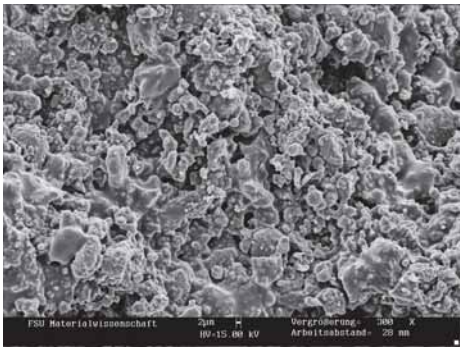

Figure 12. Microstructure of TFA-MA10 sintered at 1130^{0}C/1h, (bar 2 µm)

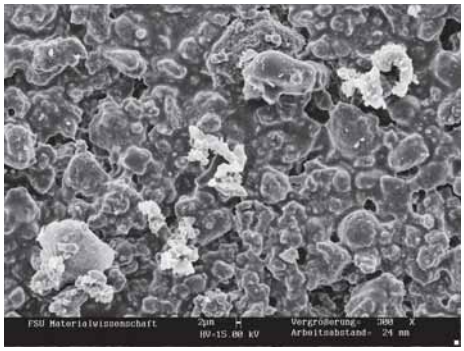

Figure 13. Microstructure of TFA-MA20 sintered at 1130^{0}C/1h, (bar 2 µm)

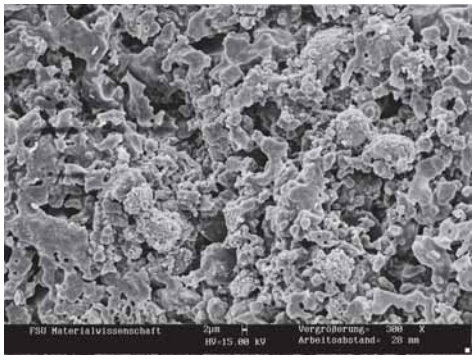

Figure 14. Microstructure of TFA-MA30 sintered at 1130^0C/1h, (bar 2 μm)

Intragranular pores with dimensions from 3 to 8 μm are present in the microstructure of the TFA-10 compact (Figure 12) and also local appearance of liquid phase is evident. The bigger quantity of liquid phase is typical for the TFA-MA20 (Figure 13) where the biggest part of the grains are covered with the liquid phase. The pores with dimensions from 4 to 8 μm are present between particular grains and non-coherent layer of the liquid phase. By increasing the time of mechanical activation to 30 min, sample TFA-MA30, the coherent part of liquids phase (Figure 13) transforms to inherent layer (Figure 14). The dimensions of the grains are from 3 to 50 μm.

4. Conclusion

In this investigation by varying the process parameters (short time of mechanical activation: 10, 20 and 30 min and sintering temperatures: 1050, 1100 and 1130^0C) a spectrum of properties is achieved. The defined optimal conditions in this investigation were a sintering temperature of 1130^0C and 20 min of mechanical activation prior to the sintering.

The obtained fly ash compacts pre-treated at 600^0C and mechanically activated for 20 min prior to the sintering at 1130^0C has the following properties: density: 2.34g/cm^3; porosity:11.46%; E-modulus: 48.04 GPa; bending strength: 79.90 MPa and compressive strength - 78.48 GPa. The compacts can be potentially used for construction purposes.

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