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Thermal stability of nano structured fly ash synthesized by high energy ball milling

J. Babu Rao[1 *], P. Narayanaswami[2] and K. Siva Prasad[3]

[1, 2] A U College of Engineering, Andhra University, Visakhapatnam, A.P, INDIA - 530 003
[3] National Institute of Technology, Trichy, Tamil Nadu, INDIA – 620 015
[*]Corresponding Author: e-mail: baburaojinugu@yahoo.com

Abstract

In this paper, an attempt has been made to modify the micro sized fly ash into nano structured fly ash using High Energy Ball Mill. The smooth, glassy and an inert surface of the fly ash can be altered to a rough and more reactive state by this technique. Ball milling was carried out for the total duration of 30 hours. The sample was taken out after every 5 hours of milling for characterizing. The nano structured fly ash was characterized for its crystallite size, lattice strain and percentage of crystallinity by using X-Ray Diffractometer. It was found that for the 30 hrs milling, the crystallite size was reduced from 92 nm to 29 nm and the percentage of crystallinity got reduced from 63% to 38%. The size, shape and texture of the fresh as well as nano structured fly ash were studied using Scanning Electron Microscopy (SEM). The fresh fly ash particles are mostly spherical in shape. The shape of the 30h milled particles is irregular and the surface morphology is rough. Isothermal annealing was carried out on 30 h ball milled fly ash from 100 to 800 ^{0}C for various times. The initial grain size of 29 nm for as-milled sample increased to 30.9 nm and 31.7 nm after annealing for 8 h at 100 and 200 ^{0}C respectively. Whereas this trend was slightly higher for higher temperatures, this increase was in the order of 29 nm to 33.43 nm, 35.35 nm, 36.53 nm, 37.78 nm, 40.42 nm, 41.93 nm after annealing for 4 h at 300, 400, 500, 600, 700 and 800 ^{0}C respectively. Hence, small crystallite size enhancement with good amount of residual strain recovery was observed during the isothermal annealing.

Keywords: Nano materials, Particulates and Powders, XRD- analysis; Fly ash.

1. Introduction

Nanoscience and nanotechnology has become the buzz-word in recent years. It has appeal of miniaturization: also it imparts enhanced electronic, magnetic, optical and chemical properties to a level that cannot be achieved by conventional materials (Bhushan, 2004; Kohar, 2004). A variety of ways have been reported to synthesize nano level materials such as plasma arcing, chemical vapor deposition, electro deposition, sol-gel synthesis, high intensity ball milling etc (Cao, 2004). Among these methods high energy milling has advantages of being simple, relatively inexpensive to produce, applicable to any class of materials and can be easily scaled up to large quantities (Baraton, 2003). In this mechanical treatment, powder particles are subjected to a severe plastic deformation due to the repetitive compressive loads arising from the impacts between the balls and the powder. This produces novel crystalline and amorphous materials with crystallite sizes at the nanometer scale (Carl, 2006).

Metal matrix composites are advanced materials resulting from a combination of two or more materials in which tailored properties are realized. It is well known that the particle- reinforced metal matrix composites have excellent mechanical properties due to the addition of the high strength and high modulus particles like TiC, Al_2O_3, SiC, TiB_2 etc. However the ductility of the MMCs deteriorates with high ceramic particle concentration (Akio *et al*., 1999). It is of interest to use nano sized ceramic particles to strengthen the metal matrix, so called metal matrix nano composites (MMNC) while marinating good ductility (Akio *et al*., 1999; Mussert, 2002). Currently, there are several fabrication methods of MMNCs, including mechanical alloying with high energy milling (Chen *et al*., 1997), ball milling (Utriga *et al*., 2003), nano sintering (Groza, 1999) vortex method (Akio *et al*., 1999), spray deposition, electrical plating, sol - gel synthesis, laser deposition etc.

Casting, as a liquid phase process, is capable of producing products with complex shapes. It will be attractive to produce as-cast lightweight bulk components of MMNCs with uniform reinforcement distribution and structural integrity. Habibnejad-Korayem *et al*. (2009) reported the enhanced properties of Mg based composites reinforced with Al_2O_3 nano particles by vortex method. A. Mazahery *et al*. (2009) developed the high performance A356 – nano Al_2O_3 composites by vortex method. Yar *et al*. (2009) studied the microstructure and mechanical properties of aluminium alloy matrix composite reinforced with nano particle MgO by vortex method. Habibnejad-Korayem et al. (2009) carried out the study on tribological behaviour of pure Mg and AZ31 magnesium alloy strengthened with Al_2O_3 nano particles by vortex method. Yang *et al*. (2004) studied the effect of nano sized SiC particles reinforced in molten aluminium alloy by ultrasonic dispersion. Lan *et al*. (2004) reported the studies on microstructure and microhardness of SiC nano particles reinforced magnesium composites fabricated by ultrasonic method.

In all the above considerations, vortex method of Aluminium based MMNCs fabrication of the nano reinforcements was added to the melt at a temperature range of 800-850 ^{0}C for the period of 30-45 minutes. Prior to addition of these nano particles, it has to be preheated for the period of 2 -3 hours at a temperature range of 700-800 ^{0}C. The grain growth and phase transformations will change the useful properties of nanostructured materials. Therefore, one fundamental requirement is that the nano sized grains and consequently the excellent properties should be retained for a desired period of time and within a significant temperature range. The grain growth of nano structured materials has been the subject of many studies in recent years (Shaw *et al*., 2003; Boylan *et al*., 1991). Zhou *et al*. (2001) investigated the grain growth in the nanostructured aluminium with an initial grain size of 26 nm. De-Castro and Mitchell (2005) investigated the grain growth in nano crystalline aluminium with initial grain sizes of 22 nm and 40 nm produced by mechanical attrition using stainless steel and nylon media respectively. No much work was available on this aspect i.e. studies on thermal stability of nano structured reinforced materials during the fabrication of MMNCs by vortex method. Hence it is worthy to carry out the some studies on thermal stability of nano structured materials to be used as reinforcements for the fabrication of MMNCs by vortex method.

There has been an increasing interest in composites containing low density and low cost reinforcements. Among the various reinforcements used, fly ash is one of the most inexpensive and low density reinforcements available in large quantities as solid waste by product from combustion of coal in thermal power plants (Kumar *et al*., 2009; Sudarshan, 2008; Ramachandra and Radhakrishna, 2007; Mondal *et al*., 2009; Daoud *et al*., 2009; Uju and Oguocha, 2009). Fly ash particles are classified into two types, precipitator and cenosphere. Generally, the solid spherical particles of fly ash are called precipitator fly ash and the hollow particles of fly ash with density less than 1.0 g/cm^3 are called cenosphere fly ash. One common type of fly ash is generally composed of the crystalline compounds such as quartz, mullite and hematite, glassy compounds such as silica glass, and other oxides. The precipitator fly ash, which has a density in the range of 2.0-2.5 g/cm^3 can improve various properties of selected matrix materials, including stiffness, strength and wear resistance and reduce the density. Cenosphere fly ash, which consists of hollow fly ash particles, can be used for the synthesis of ultra light composite materials due to its significantly low density, whch is in the range of 0.4-0.7 g/cm^3, compared with the densities of metal matrices, which is in the range of 1.6-11.0 g/cm^3 (Kumar et al., 2007; Matsunaga *et al*., 2002).

In this paper an attempt has been made to modify the fly ash by transforming the micro sized fly ash into nano structured fly ash using high energy ball mill. The smooth, glassy and an inert surface of the fly ash can be altered to a rough and more reactive state by this technique. And also some studies are carried out to assess the thermal stability of ball milled nano structured fly ash which is to be used as reinforcements for the fabrication of MMNCs by vortex method.

2. Experimental Procedure

2.1 Sample preparation

Fly Ash particles used for this study were procured from Thermal power plant of RINL (Rashtriya Ispat Nigam Limited), Visakhapatnam Steel Plant, Visakhapatnam, India. The composition of the received fly ash samples were given in Table 1. A 500 grams weight of fly ash sample was taken in a graphite crucible and allowed to preheat in the muffle furnace at 800^0 C for 3 hours to find out the loss on ignition and it was found to be 2.4%. Preheated fly ash after cooling to room temperature was washed in distilled water and removed the carbon that creamed up during washing. It was then dried at 110^0 C for 48 hours to get rid of water. Dried fly ash has been sieved for 15 minutes using BSS meshes ranging in size from 100 to 350 by Rotap Sieve shaker. Figure 1 shows the mesh size and weight fraction distribution of fly ash powder after 15 minutes sieving. The results show that more than 70% by weight retained in -200 +350 mesh; hence this size was chosen as input material for ball milling.

Table 1. Chemical composition of as-received fly ash, wt. %

SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	Loss on Ignition
58.41	30.40	8.44	2.75	1.3	1.53	1.0	1.98	2.4

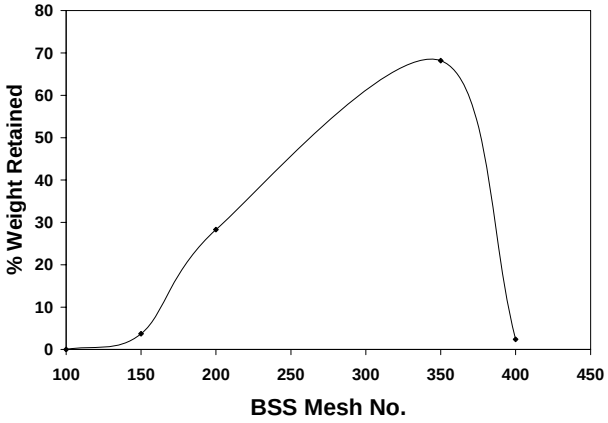

Figure 1. Distribution of as-received fly ash particles on sieving for the period of 15 minutes.

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2.2 High Energy Ball milling

The reduction in particle size of fly ash from micron level to the nano level was carried out using a high-energy planetary ball mill (Model: Retsch, PM 100, Germany) in a stainless steel chamber using tungsten carbide and zirconia balls of 10 mm Φ and 3 mm Φ ball sizes respectively. The total duration of milling was 30 hours. The rotation speed of the planet carrier was 250 rpm. The ball mill was loaded with ball to powder weight ratio (BPR) of 10:1. Toluene was used as the medium with an anionic surface active agent to avoid agglomeration. The milled sample powder was taken out at a regular interval of every 5 hours of milling. Then this powder was dried by drier and used for further characterization with an X-Ray Diffractometer (Model: 2036E201; Rigaku, Ultima IV, Japan). JADE software was used to investigate the structural changes and phase transformations of powders that occur during mechanical milling. The X-ray diffraction measurements were carried out with the help of a Goniometer model 2036E201 using Cu K_α radiation (K_α= 1.54056 A^0) at an accelerating voltage of 40 kV and a current of 20 mA. The samples were scanned in the range from 2^0 to 90^0, 2-θ and analyzed for crystallite size, peak height, crystallinity and also amount of induced strains in the milled fly ash. Scanning electron microscopy (Model: SEM – Quanta 400, FEI -Netherlands) with EDAX energy dispersive X-ray spectroscopy (EDS) was used in order to evaluate the morphological changes and the elemental analysis of certain phases observed in the nano structured fly ash powder particles.

2.3 Isothermal Annealing of Nano structured fly ash

The as-milled fly ash samples were isothermally annealed at a temperature range of 100 and 200 ^{0}C for 8 hours and from 300 to 800 ^{0}C for 4 hours and cooled in air. An X-Ray Diffractometer (Model: 2036E201; Rigaku, Ultima IV, Japan) with JADE software was used to investigate the structural changes and phase transformations of powders during mechanical milling and subsequent heat treatment. The X-ray diffraction measurements were carried out with the help of a Goniometer model 2036E201 using Cu K_α radiation (K_α= 1.54056 A^0) at an accelerating voltage of 40 kV and a current of 20 mA. The samples were scanned in the range from 2^0 to 90^0, 2-θ and analyzed for crystallite size, peak height, crystallinity and also amount of induced strains in the milled fly ash.

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3. Results and Discussion

3.1 Crystallite size and Lattice strain

Figure 2 shows the scanning electron micrograph of the fresh fly ash after heat treatment. From this figure it is evident that majority of the fly ash particles are spherical in nature and precipitator type fly ash. The morphology of fly ash particle is controlled by combustion temperature and cooling rate at the thermal power plant. The X-ray diffraction (XRD) pattern of fly ash in as-received condition is shown in Figure 3, which shows the phases present in the fly ash and are largely Silica (SiO_2), Alumina (Al_2O_3), and Mullite ($3Al_2O_3.2SiO_2$).

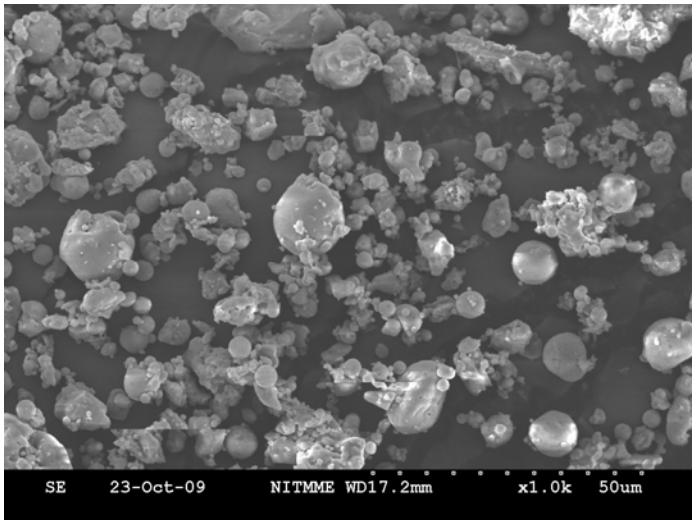


Figure 2. SEM micrograph of fly ash used in the study.

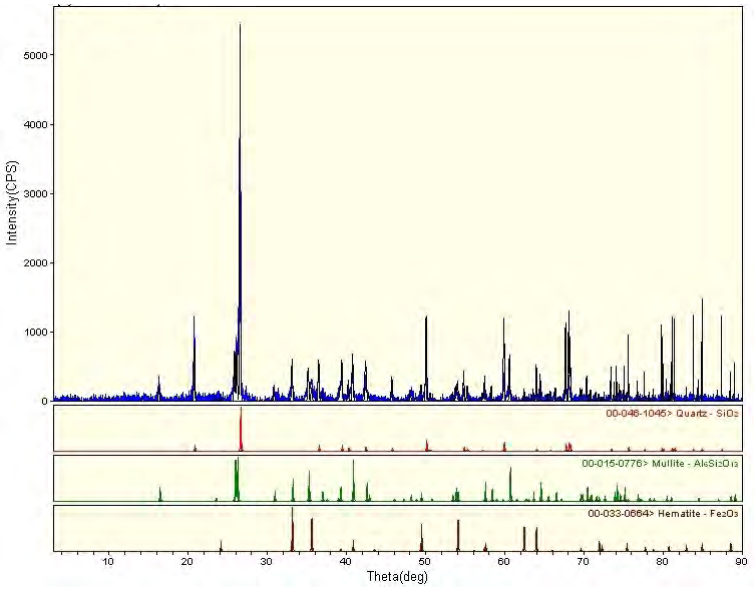


Figure 3. X-ray diffractogram of as-received fly ash.

The average crystallite size was calculated from the full width at half maximum (FWHM) of the X – ray diffraction peak using Scherer's equation (Cullity, 1978).

$$D = (k \lambda) / (B \cos\theta) \quad (1)$$

Where D is the particle diameter, λ is the X – Ray wave length, B is the FWHM of the diffraction peak, θ is the diffraction angle and K is the Scherer's constant of the order of unity for usual crystals. The existence of stress in the materials results in lattice distortions of crystals; consequently, the diffraction peaks of the crystals are broadened. The relationship between the half width of the broadened diffraction peaks, B_d and the distortion of lattice, (Δd/d) was described by Yang *et al*. (2000). The lattice distortion (Δd/d) can be obtained from the following equation.

$$(\Delta d/d) = B_d / (4 \tan\theta) \quad (2)$$

Where B_d, is half width of the broadened diffraction peaks, θ is half of the diffraction angle.

Figure 4 illustrates the variation in crystallite size and lattice strain of the fly ash with the milling time. A steady decrease in the crystallite size is observed and it was found that the crystallite size get reduced from 92 nm to 29 nm for 30h milling time. The relative lattice strain is increasing with increasing the duration of milling time. This lattice strain was increased from 0.03% to 0.36% for as-received and 30 h milled fly ash respectively. During ball milling the intense mechanical deformation experienced by the fly ash powder leads to generation of lattice strains, crystal defects and this plus the balance between cold welding and

fracturing operations among the powder particles is expected to affect the structural changes in the powder. The measurement of crystallite size and lattice strain in the mechanically milled powders is very important since the phase constitution and transformation characteristics appear to be critically depending on them Park *et al*. (2004).

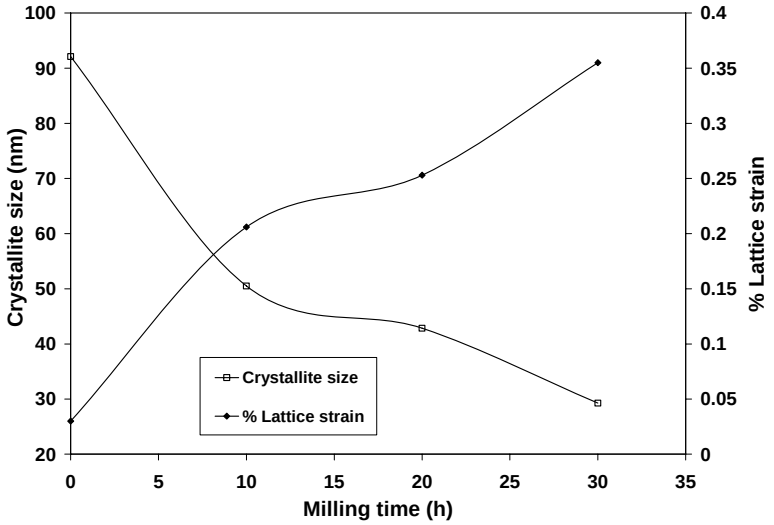
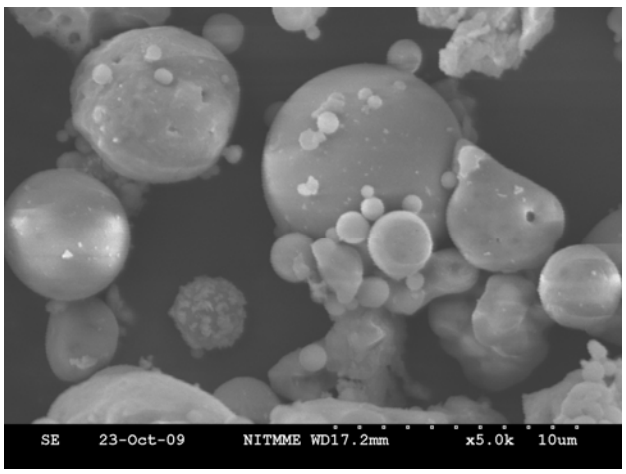
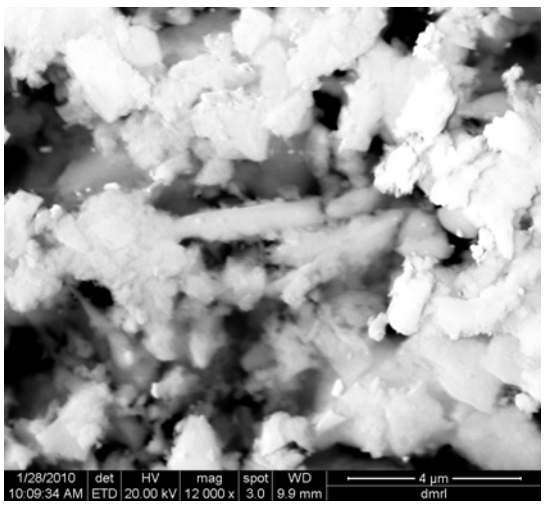


Figure 4. Variation of crystallite size and relative strain as function of milling time.

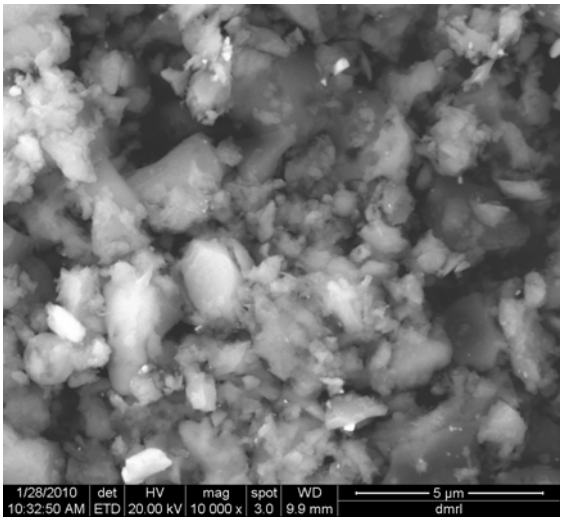
The size, shape and texture of the fresh as well as nano structured fly ash were studied using Secondary Electron Imaging mode of Scanning electron microscopy (SEM). Figure 5 (a) shows the SEM image of fresh fly ash. The fresh fly ash particles are mostly spherical in shape. The morphology of fresh fly ash particle is controlled by combustion temperature and cooling rate in the thermal power plant. Figure 5 (b) shows the SEM image of 10h ball milled fly ash. Here the spherical structure of fresh fly ash has been destroyed; and in this 10h milling stage the fly ash is in cold welding condition hence the particles appears like enlarged flakes. The large flake shaped particles are further crushed by intense impacts of the balls; hence the decrease in particle size occurs with increasing milling time as shown in Figure 5 (c, d & e), the SEM images for 20h, 25h and 30h ball milling times respectively. The shape of the particles is irregular and the surface morphology is rough. In order to determine the chemical composition of the nano structured fly ash particles for 30h milled powder, energy dispersion spectrum (EDS) was used. The Figure 6 shows the EDS spectrum of nano structured fly ash. It shows the presence of Al, Si and Oxygen peaks correspond to the Mullite (Al_2O_3. SiO_2) and Silica (SiO_2) which is present in the fly ash even after micro sized fly ash converted to nano structured fly ash.



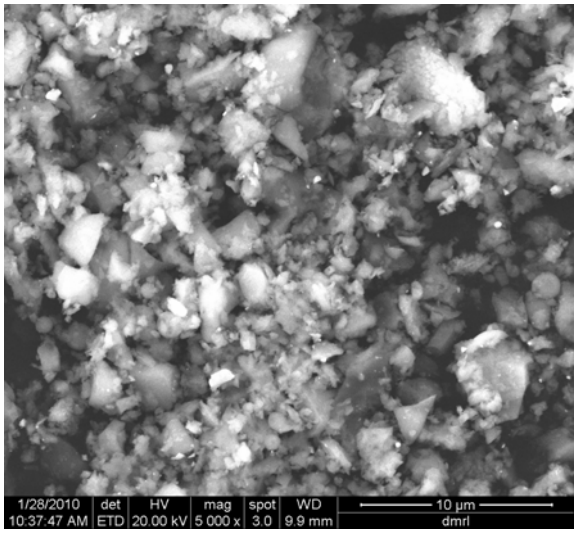
(a)



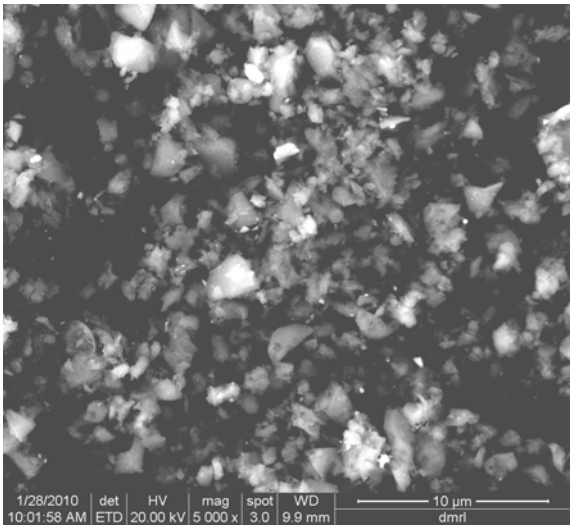
(b)



(c)



(d)



(e)

Figure 5. The scanning Electron microscope photographs of fresh and milled fly ash:
(a) Fresh fly ash (b) Ball milled fly ash for 10 h (c) Ball milled fly ash for 20 h (d) Ball milled fly ash for 25 h (e) Ball milled fly ash for 30 h.

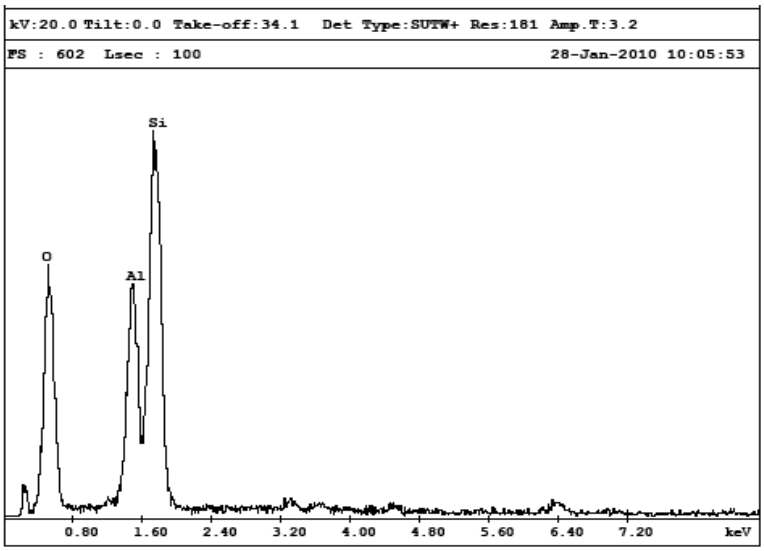


Figure 6. EDX analysis of 30 h ball milled fly ash.

The X-Ray diffractograms of the fresh as well as ball milled fly ash were shown in Figure 7 (a) and (b). Figure 7 (a) shows the 30 h milled sample.

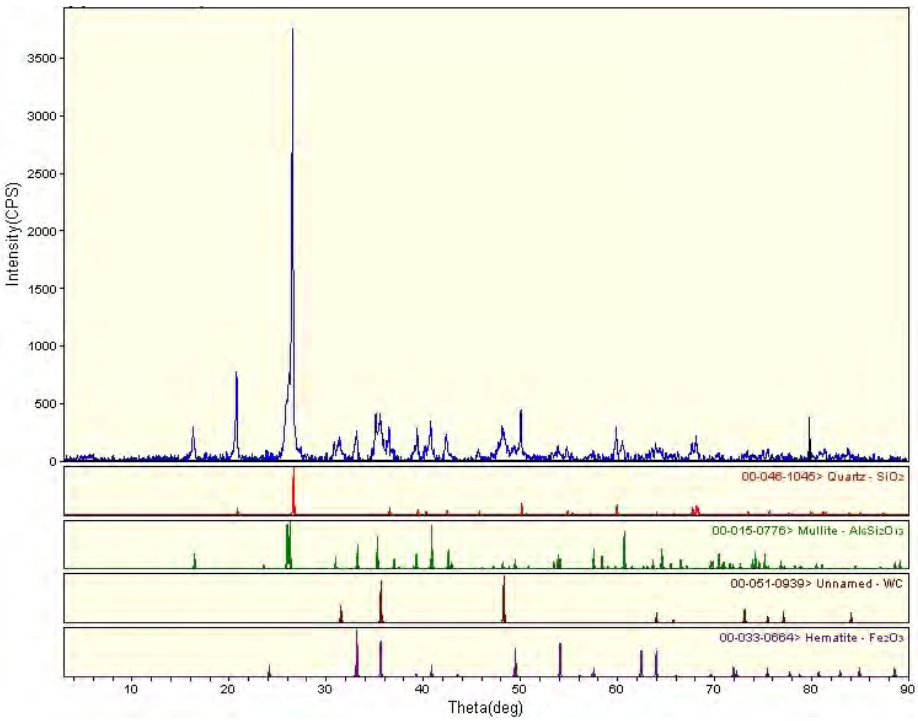


Figure 7 (a). X- Ray diffraction patterns of 30 h ball milled fly ash.

From this figure, there was a confirmation of small tungsten carbide contamination in the milled fly ash sample. This entry might be from the tungsten carbide balls which were used as milling media during milling. The X-Ray diffractograms of the fresh as well

as ball milled fly ash were shown in Figure 7 (b). From these figures, it could be clearly observed that the intensity of the sample got reduced and the peak broadening increased as the duration of milling increases; the same was represented for quartz phase in Figure 8. Three major phases were identified for all the milling times; which is Quartz (Silica), Mullite (Alumino Silicate) and Iron Oxide. Quartz phase exhibits strong peaks at 20.82^0, 26.60^0, 36.52^0, 50.08^0, 59.91^0 and 68.10^0 2θ values of (101) plane (d spacing of 4.2631, 3.3483, 2.4584, 1.8198, 1.5425 and 1.3757 A^0). Mullite phase shows strong peaks at 16.39^0, 26.16^0, 35.19^0, 41.15^0 and 60.61^0 2θ values of (210) plane (d spacing of 5.4016, 3.4034, 2.5477, 2.1915 and 1.5264 A^0). Iron oxide phase shows a peak at 24.19^0, 33.16^0, 49.74^0, 54.84^0 and 64.05^0 2θ values of (104) (d spacing of 3.6764, 2.6993, 1.8313, 1.6727 and 1.4525 A^0).

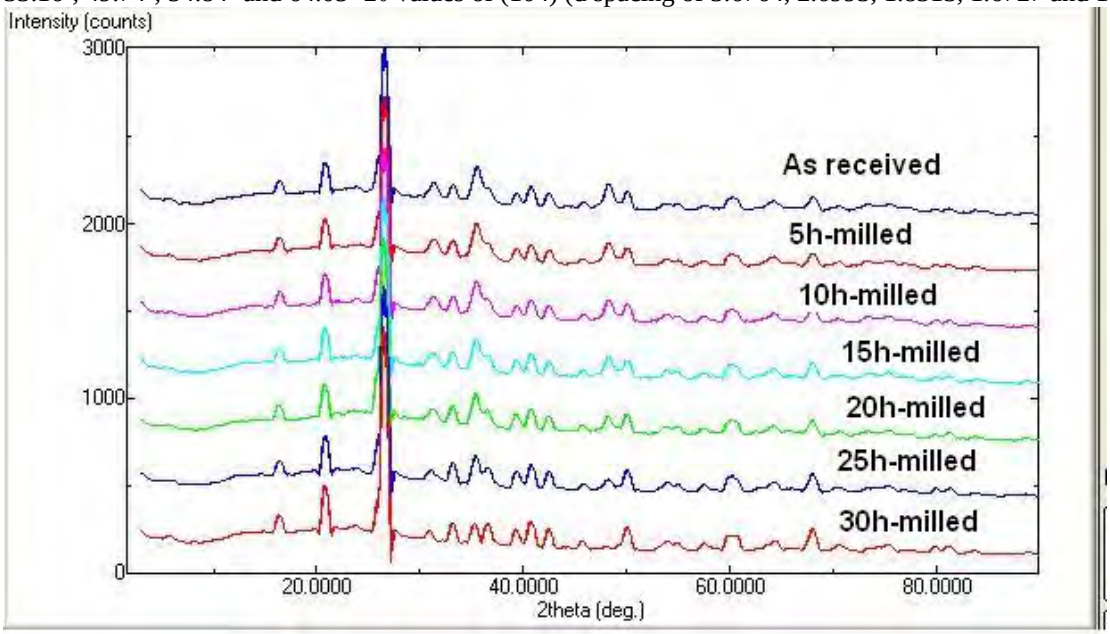


Figure 7 (b). X- Ray diffraction patterns of as-received and ball milled fly ash at different milling times.

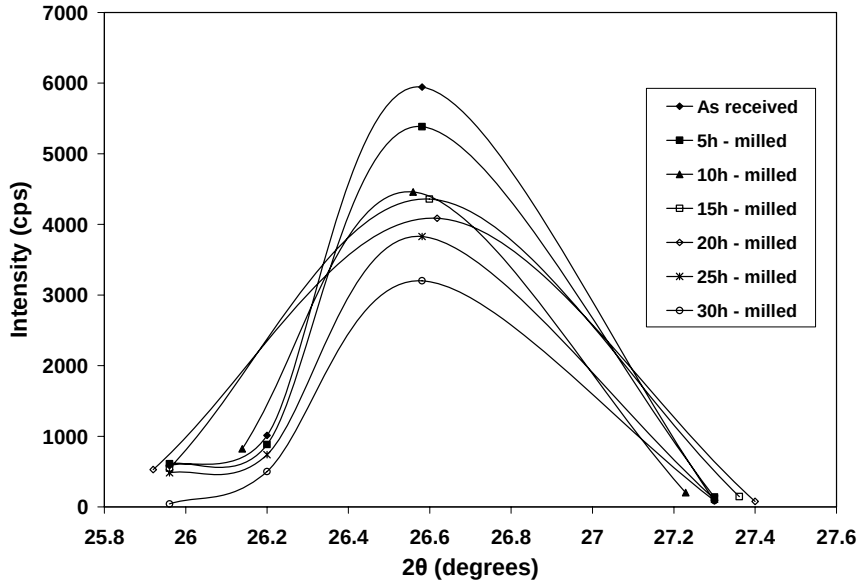


Figure 8. Displacement of SiO_2 (101) XRD peaks at different milling times.

3.2 Crystallinity

Crystallinity refers to the degree of structural order in a solid. Crystallinity is usually specified as a percentage of the volume of the material that is crystalline. The decrease in crystallinity of fly ash with ball milling hours is shown in Figure 9. This decrease was observed from 63% to 38% for fresh fly ash and 30h ball milled powder respectively. While increasing the milling time decreases the crystallinity of the fly ash, thus increasing the amorphous domains in it. This change is beneficial for the applications such as particulate nano filler in polymeric or metallic matrices. The enhanced amorphous content is very encouraging as it may lead to better compatibility with various metallic and polymeric matrices.

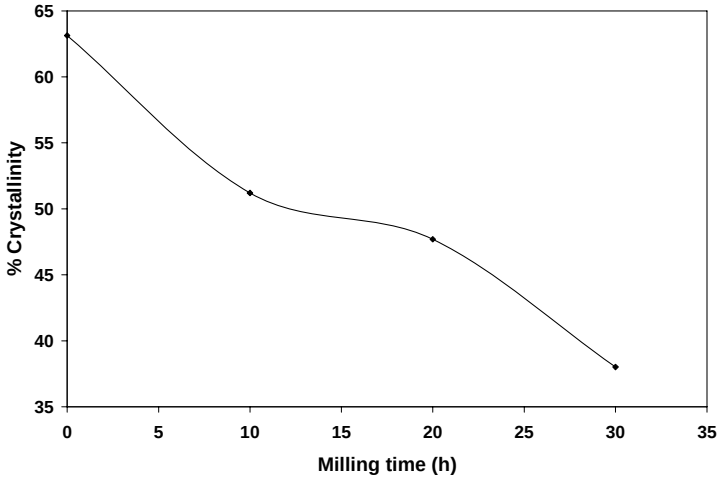


Figure 9: Variation in % crystallinity with milling time.

3.4 Structural changes of nano structured fly ash during isothermal annealing

As discussed in the previous section, 30h mechanical milled-off fly ash powders led to the formation of nano structured fly ash with a crystallite size of 29 nm and a strain level of 0.09%. This nano structured fly ash generally used as reinforcement for the production of particles reinforced aluminium based nano composites by vortex method. This process involves preheating of the reinforcements at a temperature of 800 ^{0}C for 2 hours and then dispersed into the vortex (created by mechanical stirring) of the molten aluminium alloy melt which will be held at 770 ^{0}C for the period of 15-30 minutes duration. This entire process may lead to the grain coarsening of nano structure fly ash and may also lead to the composite with micro structured fly ash reinforcements. Therefore significant structural changes can potentially occur upon heating of the as-milled fly ash powder.

Figure 10 and 11 show the variation of crystallite size as a function of annealing time at various temperatures. These figures show the milled nano structured fly ash appeared to have good stability upon annealing either for lower temperatures for longer times or higher temperature with shorter times. The initial grain size of 29 nm for as-milled sample increases to 30.9 nm and 31.7 nm after annealing for 8 h at 100 and 200 ^{0}C respectively as shown in Figure 10; whereas this trend is slightly higher for higher temperatures. This increase is in the order of 29 nm to 33.43 nm, 35.35 nm, 36.53 nm, 37.78 nm, 40.42 nm, 41.93 nm after annealing for 4 h at 300, 400, 500, 600, 700 and 800 ^{0} C respectively as shown in Figure 11. Since the ceramic particles have a lower coefficient of thermal expansion (CTE) than metallic alloys (Park *et al*., 2004; Fei and Wang, 2004; Tjong *et al*., 2003) and the thermal expansion coefficients of pure aluminium, SiC and Al_2O_3 are reported as 24.7 X 10^{-6}/ ^{o}C, 4.8 X 10^{-6}/ ^{o}C and 7.5 X 10^{-6}/ ^{o}C respectively (Xu *et al*., 1994) hence the fly ash which is composed of alumino silicates is expected to be lower CTE than either of SiC or Al_2O_3. Rohatgi *et al*. (2006) reported the CTE of fly ash cenosphere calculated as 3.3 X 10^{-6}/ ^{o}C using the rule of mixture. This low CTE of the fly ash would results the retaining of nano structured size even after annealing at either 200 ^{0}C for 8 h or 800 ^{0}C for 4 h.

Figure 12 (a-c) shows the morphology of Fly Ash particles after annealing at 200 ^{0}C for 8 hrs, 400 ^{0}C and 800 ^{0}C for 4 hrs respectively. It can be seen that the nano particles are slightly enlarged its size due to higher annealing temperatures. In order to determine the chemical composition of the nano particles, energy dispersion spectrum (EDS) was used. It is evident that Si, Al and O peaks correspond to composition of nano particles.

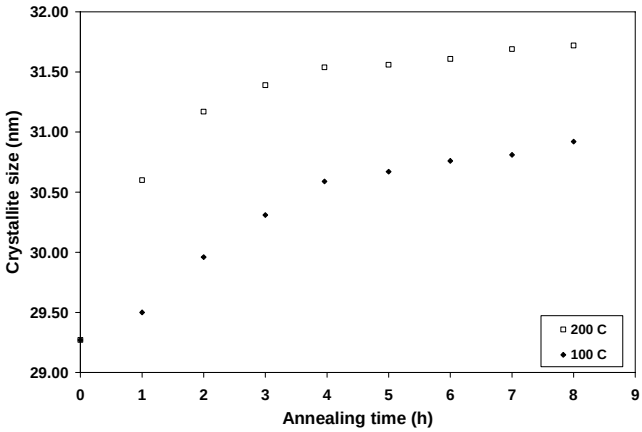


Figure 10. Variation of 30 h- milled fly ash crystallite size as function of annealing time for lower range of annealing temperature

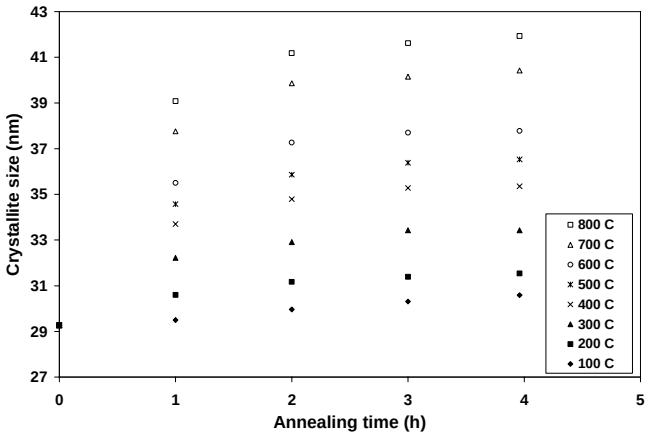
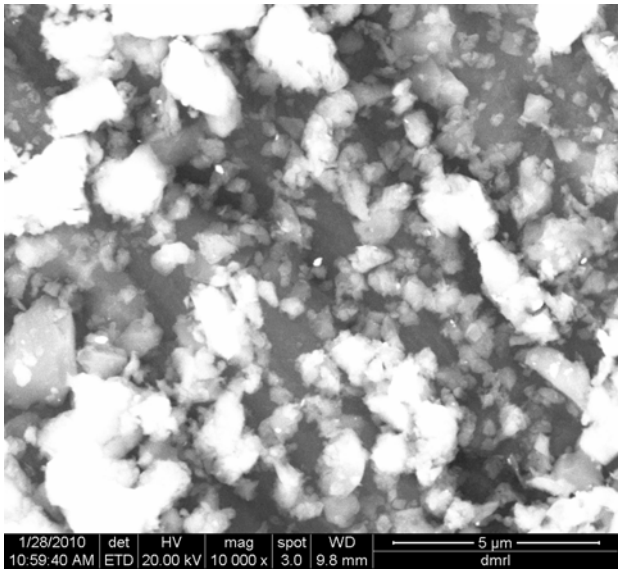
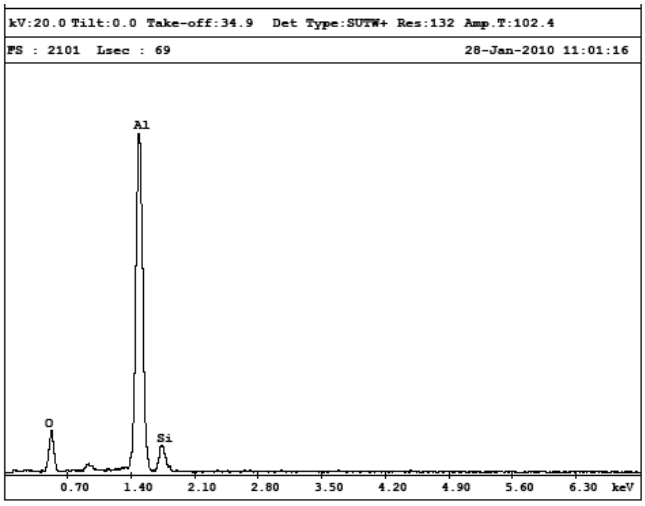


Figure 11. Variation of 30 h- milled fly ash crystallite size as function of annealing time for higher range of annealing temperature



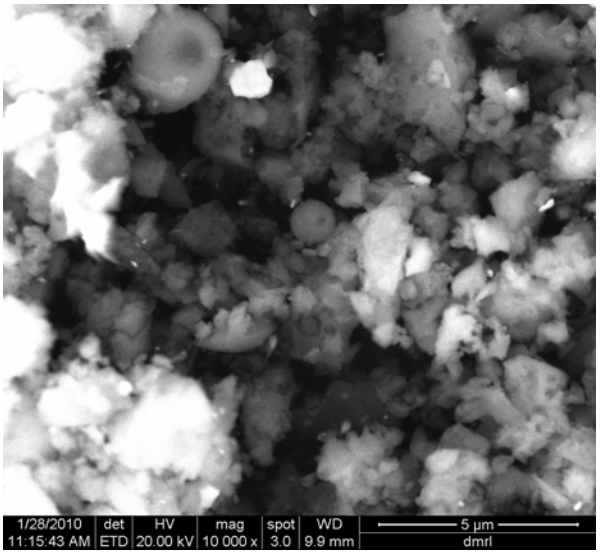


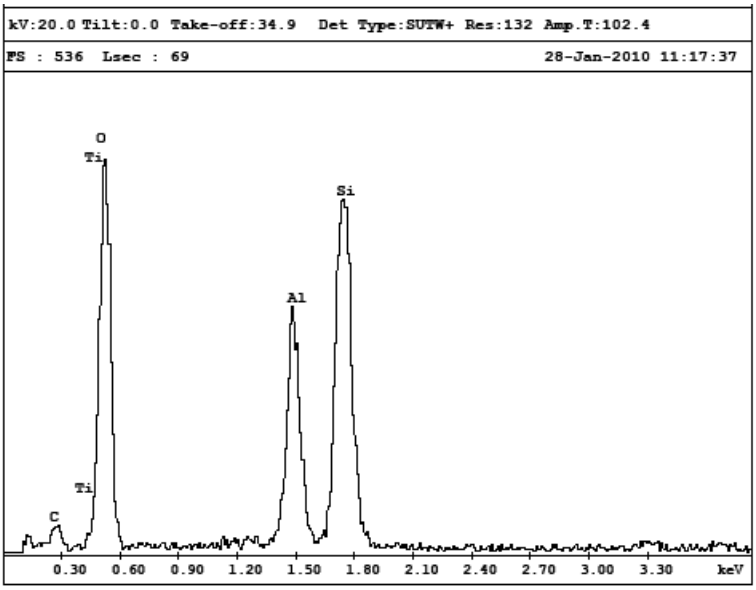
EDAX ZAF Quantification (Standardless)
Element Normalised
SEC Table : User c:\edax32\eds\genuser.sec

Element	Wt %	At %	K-Ratio
O K	24.24	35.18	0.0755
AlK	64.45	55.47	0.5411
SiK	11.30	9.35	0.0466
Total	100.00	100.00	

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
O K	16.11	0.46	3.08	34.78
AlK	165.81	0.92	0.94	179.03
SiK	13.31	0.91	3.52	14.60

(a) 200 ^{0}C – 8 hrs

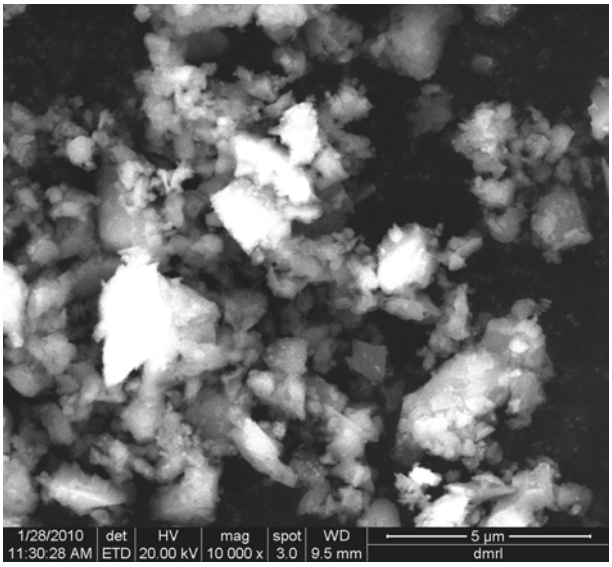


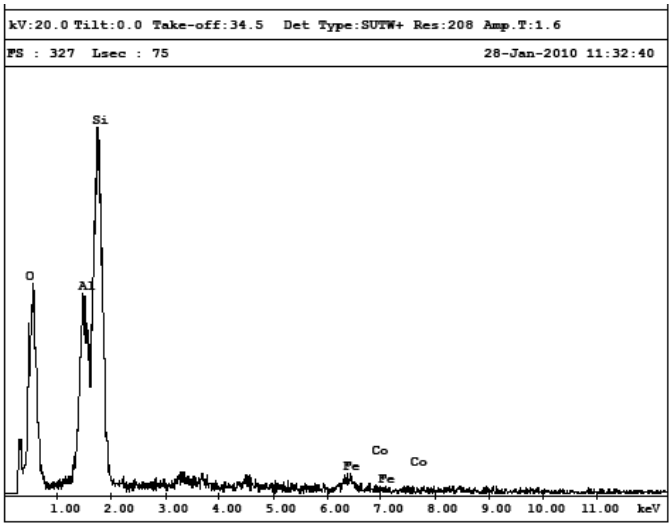


EDAX ZAF Quantification (Standardless)
Element Normalised
SEC Table : User c:\edax32\eds\genuser.sec

Element	Wt %	At %	K-Ratio
C K	13.70	20.23	0.0233
O K	52.83	58.54	0.1731
AlK	11.16	7.33	0.0753
SiK	21.60	13.64	0.1479
TiK	0.71	0.26	0.0060
V K	0.00	0.00	0.0000
Total	100.00	100.00	

(b) 400 ^{0}C – 4 hrs





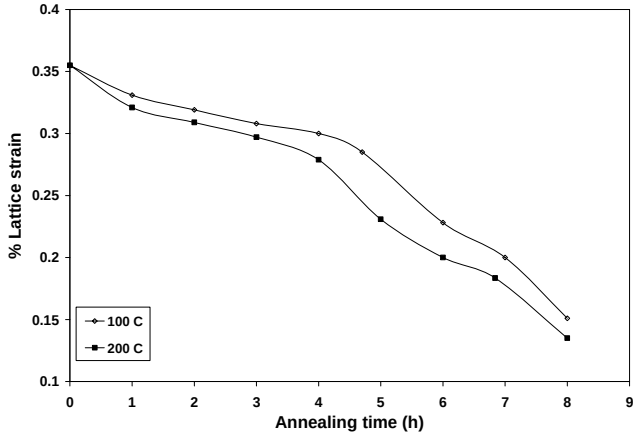
EDAX ZAF Quantification (Standardless)
Element Normalised
SEC Table : User c:\edax32\eds\genuser.sec

Element	Wt %	At %	K-Ratio
O K	44.47	59.34	0.1585
AlK	15.61	12.35	0.1056
SiK	34.61	26.30	0.2235
FeK	4.23	1.62	0.0369
CoK	1.08	0.39	0.0092
Total	100.00	100.00	

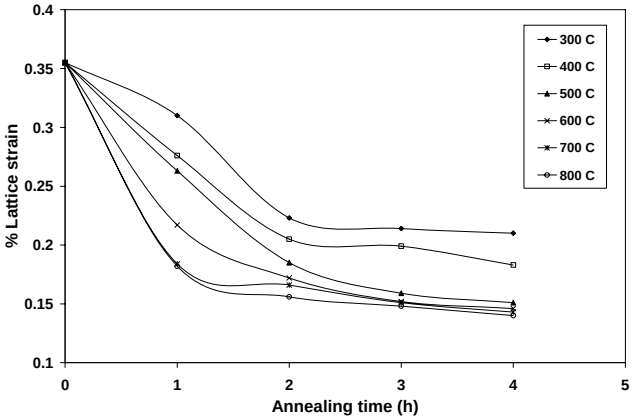
(c) 800 ^{0}C – 4 hrs

Figure 12. The scanning Electron microscope photographs with EDS analysis of 30h milled and annealed for 4 hrs at different annealing temperatures: (a) 200 ^{0}C – 8 hrs (b) 400 ^{0}C – 4 hrs (c) 800 ^{0}C – 4 hrs

Figure 13 (a & b) shows the 30h milled nano structured fly ash powders annealed for various annealing times at different annealing temperatures of 100 and 200 ^{0}C for 8 h and 300-800 ^{0}C for 4 hours to asses the recovery of lattice strain in the milled powder. If the temperature is high, the associated higher diffusivity (higher atomic mobility) leads to processes resulting in recovery and recryastallization; on the other hand, if the temperature is low then strain recovery would be less (Koch, 1993). So that the initial strain of 0.355% for as-milled sample decreases to 0.151% and 0.135% after annealing for 8h at 100 and 200 ^{0}C respectively as shown in Figure 13 (a); whereas this trend is slightly higher for higher temperatures. This decrease is in the order of 0.355% to 0.21%, 0.183%, 0.151%, 0.146%, 0.143% and 0.14% after annealing for 4 h at 300, 400, 500, 600, 700 and 800 ^{0} C respectively as shown in Figure 13 (b). For the longer annealing times and higher annealing temperature show the way to the very low strain levels in the milled powder.



(a)



(b)

Figure 13. Internal residual strain of 30 h - milled nano structured fly ash as function of annealing temperature for various annealing times (a) Low range of annealing temperature (b) High range annealing temperature.

Figure 14 shows the XRD patterns of the 30 h – milled fly ash after annealing at different temperatures for 4 h.

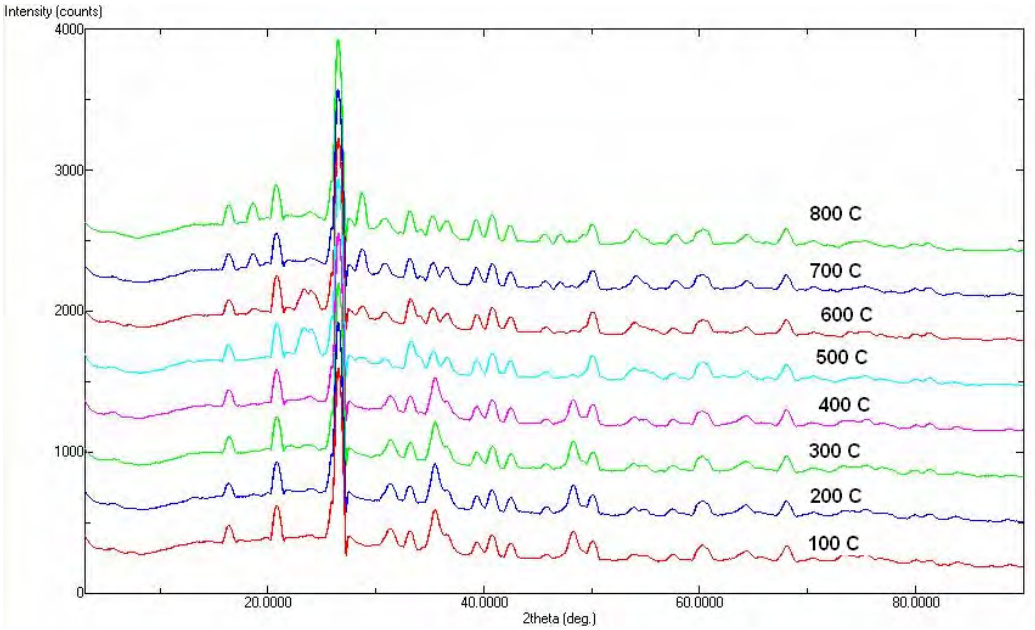


Figure 14: XRD patterns of 30 h milled Fly ash powder particles after annealing for 4 h at various temperatures

Three major phases were identified for all the annealing temperatures; which are Quartz (Silica), Mullite (Alumino Silicate) and Iron Oxide and also small amount of Tungsten Carbide (WC) was present as contamination. At 500 ^{0}C tungsten oxide (WO_3)

appeared in the fly ash particles; which might be due to the reaction between Tungsten carbide (WC) and oxygen from the oxides of either Silica, Mullite or from Iron oxide. At 700 ^{0}C WO_3 was converted to WO_4.

4. Conclusions

The size reduction of fly ash from micrometer level to nano meter levels has been achieved by high energy ball milling for the period of 30 hrs. The percentage of crystallinity reduces from 63% to 38%. The fly ash becomes more amorphous and the crystallite size reduces drastically. The lattice strain was increased with increasing the milling time. The SiO_2 phase is the maximum sufferer during milling; hence with increasing the milling time SiO_2 peaks shifts slightly to the lower angels and also broadening of the diffraction pattern occurs. Small crystallite size enhancement with good amount of residual strain recovery was observed during the isothermal annealing process. By and large, nano structure still exists in the 30h ball milled fly ash even after the isothermal annealing. Currently, the author is utilizing this nano structured fly ash for synthesis of Aluminium- fly Ash metal matrix nano composites (MMNC). The work is on progress and the results will be presented in the next publication.

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Biographical notes

Dr. Babu Rao Jinugu, working as Associate Professor in the Dept. of Metallurgical Engineering, AU College of Engineering, Andhra University, Visakhapatnam since May 2000. He completed his B. Tech and M. Tech in Metallurgical Engineering from NIT Warangal and Ph. D from Andhra University, Visakhapatnam. Prior to join in this University, he worked as Assistant Manager (QA & TD), R&D division of RINL, Visakhapatnam Steel Plant, Visakhapatnam since Sept'1995-April 2000 and also worked as Scientist in C-MET Laboratories, Dept of Electronics, Hyderabad for a period of two months (August - September 1995). He is the Life member for The Indian Institute of Metals (IIM) – Kolkata, Life Member for Tribology Society of India (TSI) and Life member for The Institution of Engineers (IEI). At present he is holding the positions of Joint Secretary - IIM Visakhapatnam Chapter, Chairman - IIM Visakhapatnam Students Chapter and Member- Editorial Board of IIM News Magazine. Currently working for UGC, New Delhi sponsored Major Research Project (File No: 34 -396/2008 (SR) on "Synthesis and Characterization of Al-Fly Ash Nano Composites" and also working for one AICTE, New Delhi sponsored research project (File No: 1-51/FD/CA/(19)2006-2007) on "Studies on cold workability limits of Aluminium and its alloys". He was chosen by AICTE-New Delhi as "Career Award for Young Teacher" in the year 2007. He has visited Beijing, China and Singapore in the year 2008 for participation and presenting the papers at various international conferences. At present, a total of 53 Research Papers were published at various national and International journals/ conferences. His current areas of research are Nano Composite Materials and Metal Forming.

P Narayanaswami is a student of M Tech (nano Technology) in the Dept of Metallurgical Engineering, AU College of Engineering, Andhra University, Visakhapatnam, India.

Dr. K Siva Prasad working as Assistant Professor in the Dept of Metallurgical and Materials Engineering, National Institute of Technology, Trichy, Tamil Nadu, India. He obtained his M Tech and Ph D from the Indian Institute of Technology, Madras, Chennai, India. He is author of more than 50 national and international Journals.

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