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SYNTHESIS AND STRENGTH STUDY OF CEMENT MORTARS CONTAINING SiC NANO PARTICLES

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In this work we have demonstrated a series of experiments for the synthesis of nano scale silicon carbide (SiC) crystallite in cement mortar in powder form and studied the stress-strain (and strength) of cement mortar containing SiCnano particles using AFM (Atomic Force Microscopy)and XRD (X-ray Diffraction) techniques and X- powder, Williamson-Hall andNanosurf methods.The obtained results show a more stable structure of the sample with 10% SiCnano particles.

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Keywords: Cement, SiC, Nanoparticles and AFM technique.

1. Introduction

Many current strategies and investigations for material synthesis integrate both synthesis and assembly into a single process, such as characterizes mechanical, physical and chemical synthesis of nanostructuralmaterials [1-3].Major efforts in cement mortar powder nanocrystallite and nanoparticle synthesis can be done using sol- gel methods.

Furthermore, nanocompositematerials have shown many potential applications due to their functionality, their chemical reactivity and/or physical compactionto integrate nanostructure building blocks within the fine final material structure [4-9].

In the last decade, many workers have studied the high performance multifunctional cementitiousnanocomposite materials with different types of nanoparticles[11], mostly Si, Co, C, Fe and nanoclayparticles [11 and references therein]. However, their concrete samples containing above nanoparticles are less resistant to bending than samples with SiCnanopparticlesinthe present work.

We firstly synthesized the SiC nanoparticles using the sol-gel method at different temperatures and nanoparticles molarity. After that we dispersed these nanoparticles in a cement solvent and sonicatedthe mixture. The different samples have been prepared and tested during two weeks to detect variation incompression, bending and contraction properties as studied using AFM and XRD techniques and evaluated with X- powder, Nanosurf and Williamson- Hall, methods. The obtained results showed an increase in the resistance to bending for 3% SiCnano particles concretein comparison with undoped concrete.

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2. Experimental Procedures and Details

Mortar cement is produced by the usual procedure [10]. The samples, nanometer silicon carbide powders, have been synthesized with the sol- gel method and carbothermal reduction processing with TEOS (tetraethoxysilane), $(C_2H_5)_4SiO_4$) and saccharose ($C_{12}H_{22}O_{11}$) as starting materials.This method is a wet chemical synthesis approach for generating nanocrystallitesand/ ornano cement mortar particles with SiCcontents by gelation, precipitation and hydrothermal treatment [12].

At first, Silica aqueous solutionwas prepared by adding 10 g of saccharose ($C_{12}H_{22}O_{11}$) to 600 ml of de-ionized water at room temperature and vigorously stirring for 4 hours on hotplate with magnet. Then, ethanol (CH_3CH_2OH) as cosolvent and hydrochloric acid as catalyst were added dropwise into the wellstirred Silica aqueous solution. It further dehydrated to make colorless and transparent gel and dried to obtain drying gel at 80˚C. When addition was complete, stirring was continued for 24 h at 80 ˚C. The final powder of SiCnanocrystallites was obtained by drying the precipitation at room temperature and 300 ˚C for 48 h.

The surface morphology and crystal structure of nanometerSiC powders have been investigated using XRD, AFM, Nanosurf and X- Powder techniques. The crystal phases of the nanocrystallites were identified by XRD analysis (figures 1 and 2) and the nanocrystallitessizes were also appraised by X-powder method. The crystal phases of the SiCnanocrystallites were identified by XRD analysis. The Miller index in figure 1 is not clear as in figure 2 due tocement structure.

The SiCnanocrystallitesize (corresponding to figure 1) was appraised by X-powder method. The typical diameter foundfor 3% SiCnanoparticles andundoped cement were 48 nm and 29 nm, respectivly. X- Powder technique (figures 3 and 4 for SiC crystallite phase) shows that typical diameters reach nanometer magnitude. The smaller size of cement with 3% SiCnanoparticleswith respect to that of undopedcement, indicates that temperature and SiC affect the crystallites structure.Experimental results show that the samples have better crystalline state with 3% SiCnano particles. As shown in figure 2 (down), a relatively amorphous structure could be formed at room temperature, as well.

Microscopy and surface morphology analyses were performed by using AFM (figures 5 and 6) and Nanosurf techniques(figures 7 through 10). AFM topography images of the SiCnanocrystallites at pH = 8 and the image of height distributions of SiCnanocrystallites at pH = 8 are shown in figures 11 and 12, respectively.

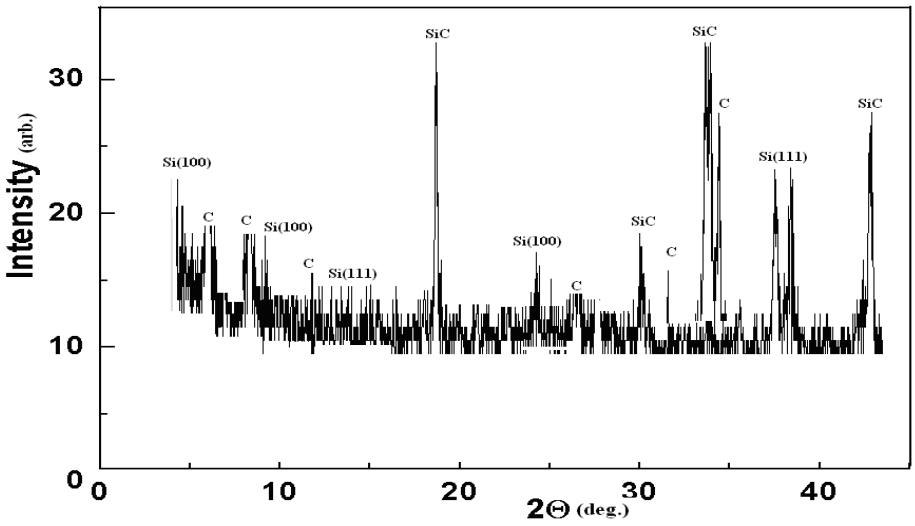

Fig. 1.The crystal phases of the mortar cement nanocrystallites were synthesized at 300^{0}C and identified by XRD analysis. The Miller indicesare not clear due to the cement structure.There are no significant peaks after 40^o and we eliminated these peaks.

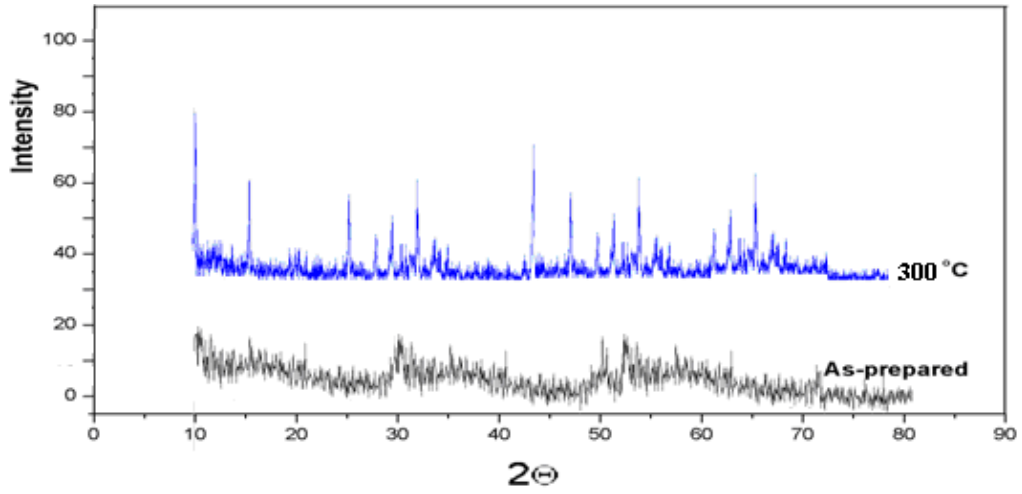

Fig. 2.The crystal phases of the cement mortar with 3% SiCnanocrystallites were synthesized at room temperature (down) and 300^{0}C (up) and identified by XRD analysis.

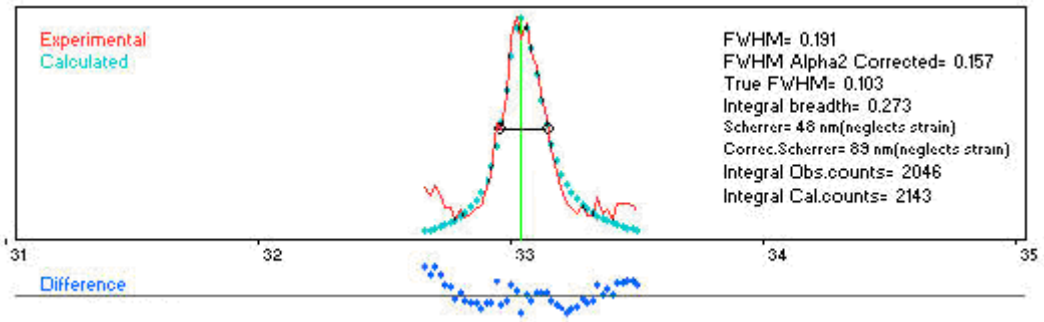

Fig. 3 -The size of SiCnanocrystallite (corresponding to figure 1) is appraised by X-powder method. It is shown that the typical diameter is of 48 nm.

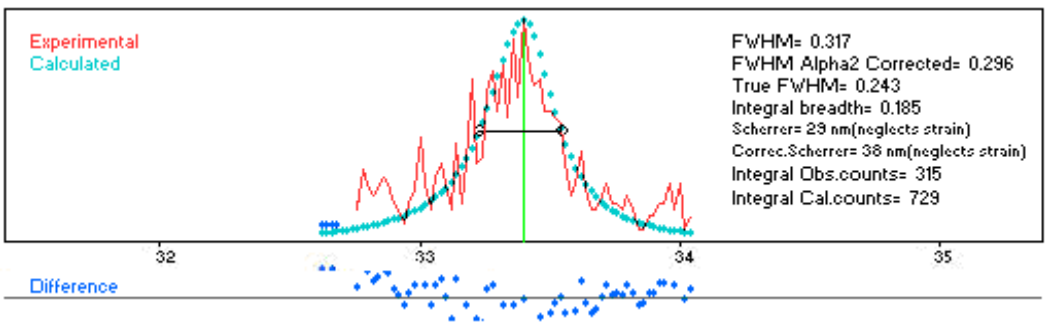

Fig.4- The size of SiCnanocrystallite (corresponding to figure 2 at 300^{0}C) is appraised by X-powder method. It is shown that the typical diameter is of 29 nm.

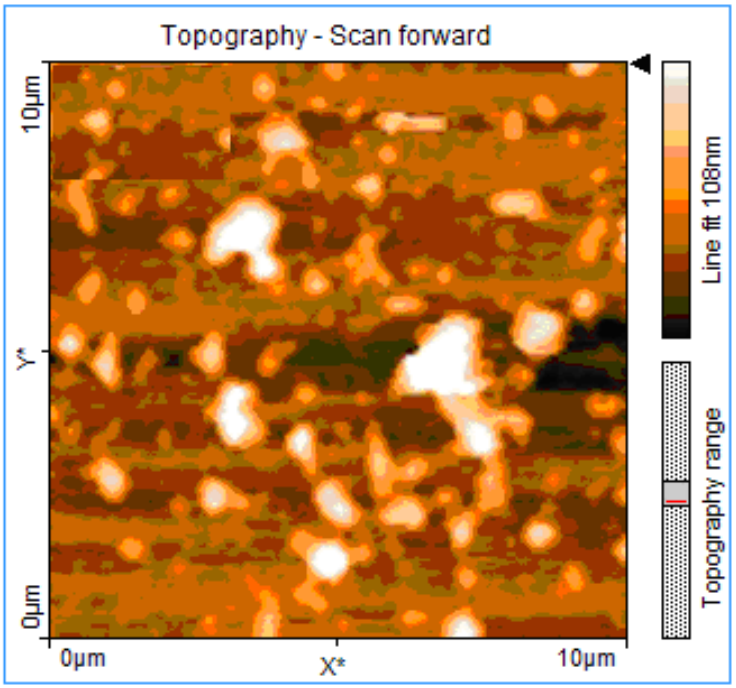

Fig. 5 -Microscopy and surface morphology analyses of cement prepared at 300^{0}C and performed by using AFM technique.

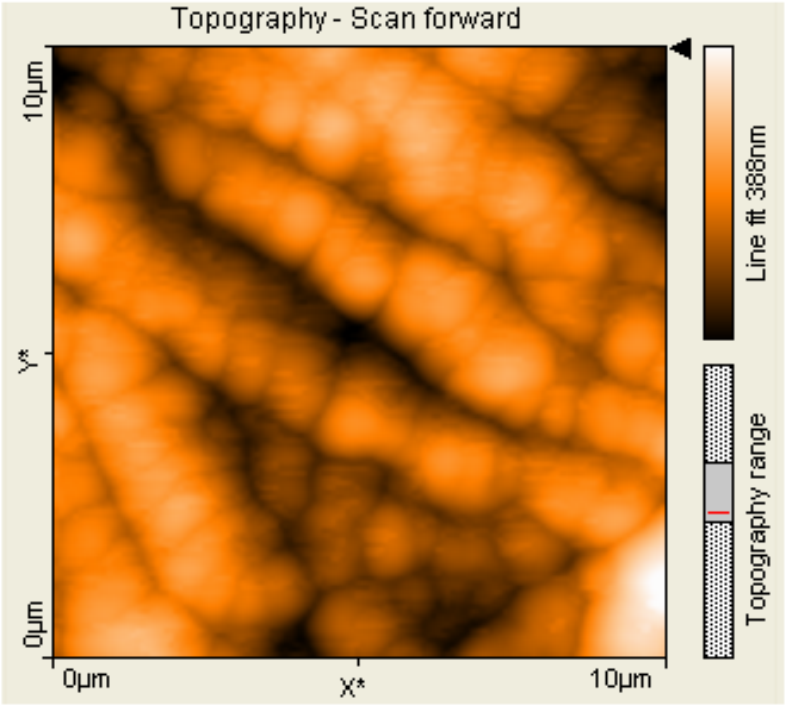

Fig. 6.Microscopy and surface morphology analyses ofcement mortar with 3% SiCnano particles prepared at 300^{0}C and performed by using AFM technique.

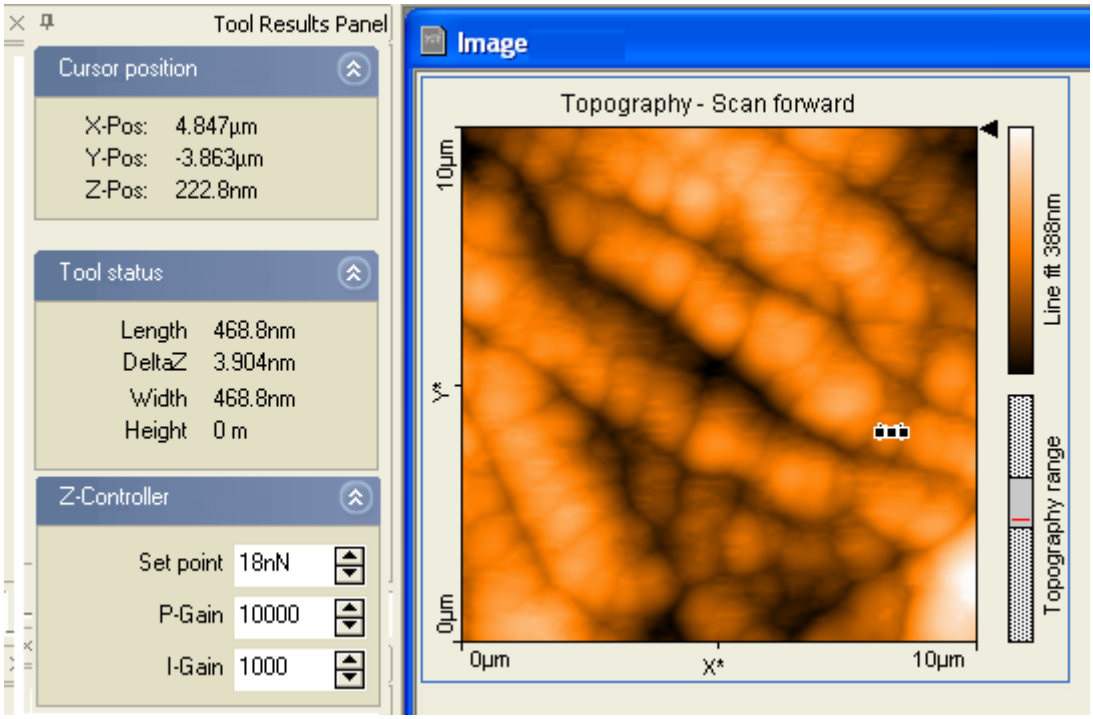

Fig. 7.Dislocation study of thecement mortar with 3% SiCnano particles prepared at 300^{0}Cby using the Nanosurf (see closed square) technique and the results of stress and strain are shown in the left hand side of the AFM image. The SiC crystallite length of the cement is 468.8 nm.

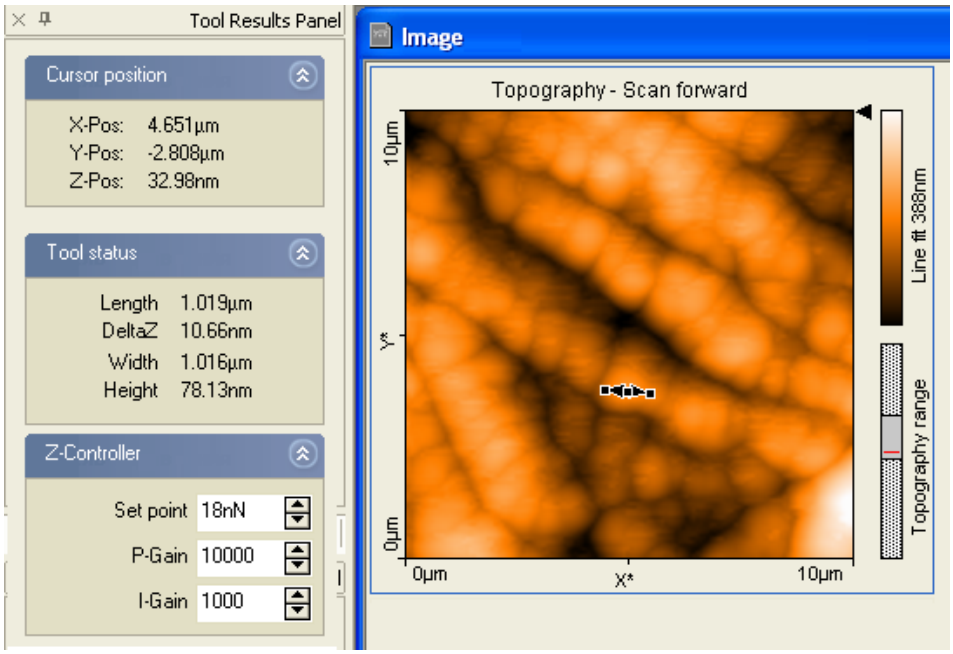

Fig. 8.Dislocation study of thecement mortar with 3% SiCnano particles prepared at 300^{0}Cby using the Nanosurf (see closed square) technique and the results of stress and strain are shown in the left hand side of the AFM image. The SiC crystallite length of the cement is 1019 nm.

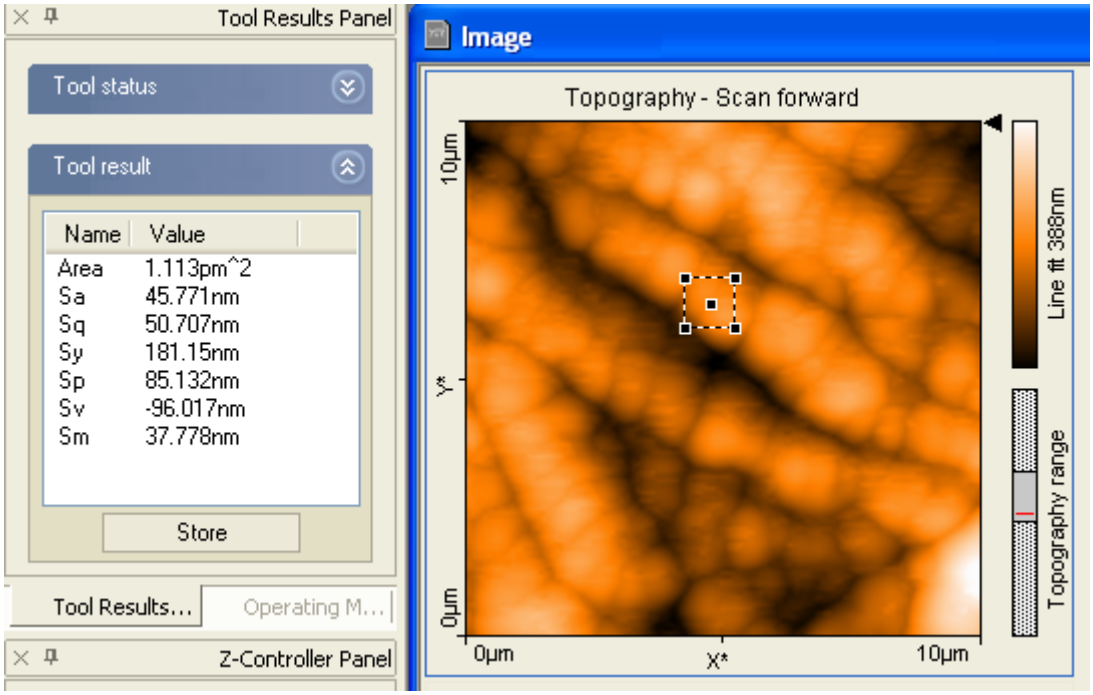

Fig. 9.Dislocation study of the cement mortar with 3% SiCnano particles prepared at 300^{0}Cby using the Nanosurf (see closed square) technique and Williamson- Hall data for stress and strain are shown in the left hand side of the AFM image.

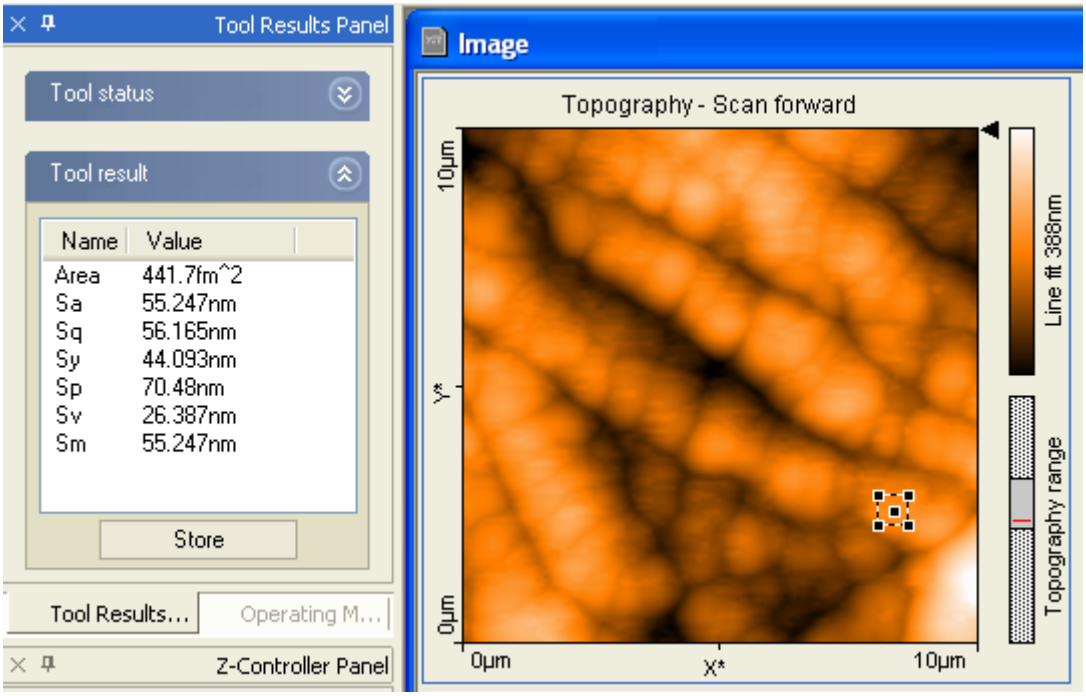

Fig. 10.Dislocation study of the cement mortar with 3% SiCnano particles prepared at 600^{0}Cby using the Nanosurf (see closed square) technique and Williamson- Hall data for stress and strain are shown in the left hand side of the AFM image.

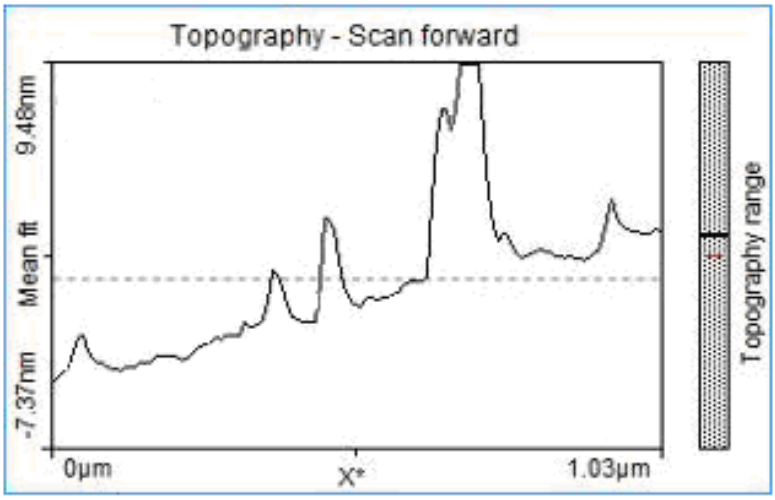

Fig. 11.AFM topographical images of synthesized cement mortar with 3% SiCnano particles at 300^{0}C at pH = 8. The scan size was 25.12 pm^2. S_a of the synthesized SiCnanocrystallites is about 7.121 nm at pH = 8.

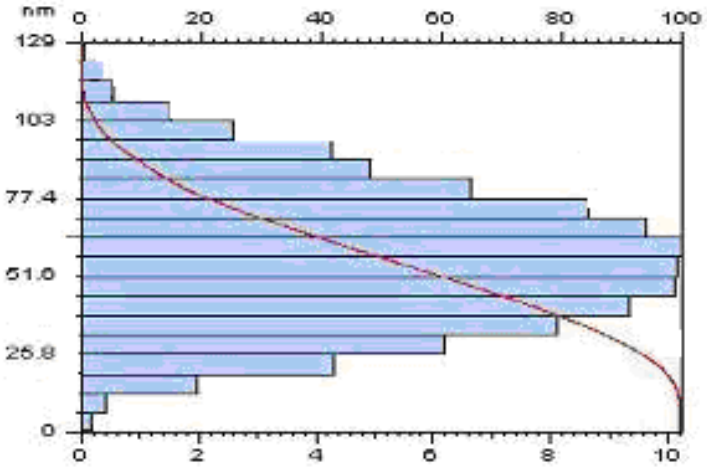

Fig.12. This image shows the height distribution of synthesized cement mortar with 3% SiCnano particles at pH = 8. It reveals that the SiCnanocrystallites at pH = 8 with tight surfaceare suitable in temperature sensor.

In addition, the present matrix is a standard silica fume and cement produced by Mazandaran, Iran agglomerated diamond Si/C. Cement mortar specimens of a polyhedron shape, 5×5×20 cm^3 in size, are manufactured and cured in wet air media. AFM topographical image in figure 11 shows synthesized SiCnanocrystallites at 300^{0}C at pH = 8. The scan size was 25.12 pm^2. S_a of the synthesized SiCnanocrystallites are given in the Nanosurf images at pH = 8. Uniform surface was observed with many nanoparticles at pH = 8. There is rougher surface, as revealed in topography spectrum in figure 11. The Gaussian distribution of mortar cement with 3% SiC nanoparticles in figure 12, indicates a homogeneous distribution of siCnano particles in the mortar cement structure.

3. Discussions

As shown in above figures and images, the large SiCnanocrystallites bounded to cement mortar matrix are as nano building blocks for nanoporous molecular sieves. These nanoparticles aggregate as polymer- like units and/ or heavy quantum wires. SiC nanoparticles as additives in concrete affect strongly concrete structure due to their chemical and mechanical properties which are different from those of individual primary particles. The size of SiCnanocrystallites is measured by using both X- powder and Nanosurf techniques and the results are inserted figures 3, 4 and 7, 8. Depending on the particle size and its compositional material, the bonding force responsible for holding the SiC nanoparticle aggregates together varies from weak Vander Walls force for micrometer particles to strong chemical bonds for nanometer particles to very strong electrical dipolar bonds for nanosizedSiC particles.

The stress- strain diagrams of the samples are measured with theNanosurf technique andWilliamson- Hall formula (equation1) [13], as shown in figures 13.

$$\beta cos\ = \frac{k\lambda}{L} + 4sin\theta \qquad (1)$$

Where L is the size of the nanoparticle and θ is the full- width half- maximum.

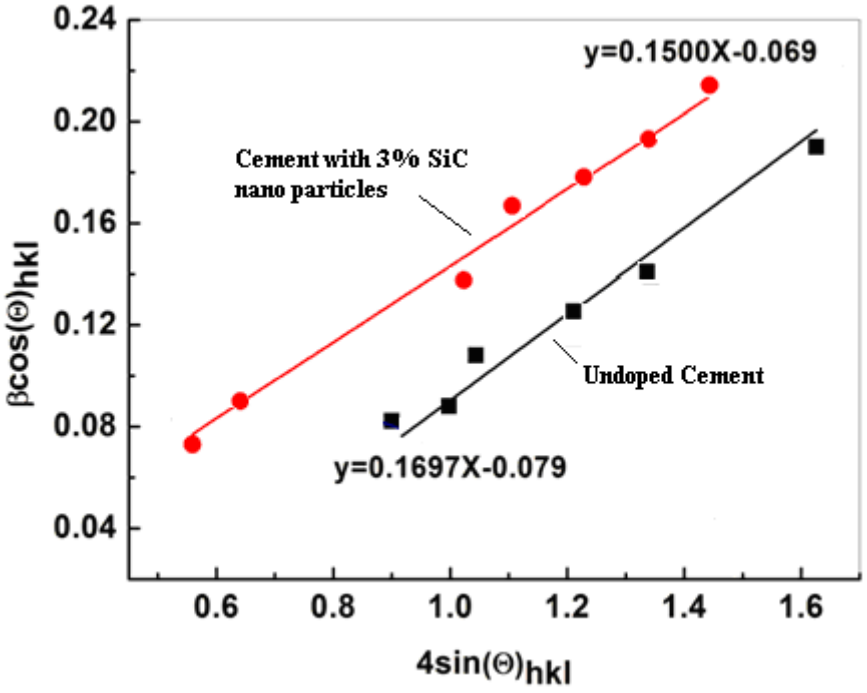

Fig. 13. The Williamson – Hall method based on X- Powder findings shows linear diagrams of βcosθ versus 4sinθ.

XRD spectra and AFM images in the above figures show that cement mortar with 3% SiCnanoparticlesis a more rigid matrix than that without SiCnano particles. The penetration of SiCnanoparticles through the cement can make much more room for nanoparticles to improve the plain cement mortar properties. The scattering spectrum in figure 12 shows that particles are uniformly dispersed. The reasoncould be due to their great surface energy.

4. Conclusion

It is clear that the temporal change of the bending strength for concrete with 3% SiCnano particles and undoped cement has shown measurable differences. In figure 13, one can see that harder and less resistant to bending samples can be produced with SiC nanoparticles.

Acknowledgment

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