

# Influence of nano-silica on the failure mechanism of concrete specimens

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(Received September 11, 2016, Revised January 10, 2017, Accepted January 13, 2017)

**Abstract.** Failure of basic structures material is usually accompanied by expansion of interior cracks due to stress concentration at the cracks tip. This phenomenon shows the importance of examination of the failure behavior of concrete structures. To this end, 4 types of mortar samples with different amounts of nano-silica (0%, 0.5%, 1%, and 1.5%) were made to prepare twelve 50×50×50 mm cubic samples. The goal of this study was to describe the failure and micro-crack growth behavior of the cement mortars in presence of nano-silica particles and control mortars during different curing days. Failure of mortar samples under compressive strength were sensed with acoustic emission technique (AET) at different curing days. It was concluded that the addition of nano-silica particles could modify failure and micro-crack growth behavior of mortar samples. Also, monitoring of acoustic emission parameters exposed differences in failure behavior due to the addition of the nanoparticles. Mortar samples of nano-silica particles revealed stronger shear mode characteristics than those without nanoparticles, which revealed high acoustic activity due to heterogeneous matrix. It is worth mentioning that the highest compressive strength for 3 and 7 test ages obtained from samples with the addition of 1.5% nano-silica particles. On the other hand maximum compressive strength of 28 curing days obtained from samples with 1% combination of nano-silica particles.

**Keywords:** failure behavior; micro-crack growth; acoustic emission; nano-silica particles

## 1. Introduction

Concrete and mortar are construction materials generally used due to their low price and high compressive strength, and enhancing their performance has been great concern of the research community (Fu and Chung 1998).

The hardening processes of concrete is caused by chemical reactions over time that occur between water and cement as the main and substantial parts of concrete. This process leads to formation of a kind of glue which makes a cohesive mass with the aggregates. The glue which gets stronger with age is called Calcium-Silicate-Hydrate (C-S-H) gel and is responsible for most of the engineering properties of cement based concrete. C-S-H gel as the main source of reaction product in concrete is made up of nanometric particles which can contribute to the strength of concrete and decrease in the porosity (Zhang *et al.* 2000, Girao *et al.* 2007, Beaudoin *et al.* 2009).

Formerly, the effects of the addition of silica nanoparticles on different mechanical properties of concrete composites have been studied. For instance, Li *et al.* (2006 and 2007) examined the enhancement of compressive and flexural strengths together with abrasion resistance and chloride permeability as well as flexural fatigue performance of nanomaterials binary blended concrete. Other researchers such as Belhadj *et al.* (2014) investigated the effects of the type of sand on the fracture and mechanical properties of sand concrete. They also revealed

that the sand having the highest diameter and the best particle size distribution has given the best fracture and mechanical properties.

In addition, Heidari and Tavakoli (2013) investigated the compressive strength as well as water absorption of binary blended concrete samples utilizing nano silica in the presence of waste ground ceramic simultaneously. They showed that the incorporation of nano silica particles can highly improve the compressive, flexural and tensile strength plus toughness of blended concrete samples. Also Zhang *et al.* (2014a and 2014b) are investigated the combined effect of nano-SiO<sub>2</sub> particles and steel fibers on flexural properties of concrete composite containing fly ash and mechanical properties of fly ash concrete composites reinforced with nano-SiO<sub>2</sub> and steel fiber which are both showed the improved properties of binary and ternary blended concrete samples. Moreover, the effect of silica fume on fresh properties, compressive strength at 28 days and fracture behavior of fly ash concrete composite were studied by Zhang *et al.* (2016) which indicated the capability of concrete composite containing fly ash to resist crack propagation. It is well known that, nanoparticles can act as heterogeneous nuclei for cement pastes, then accelerating cement hydration process due to their ultra-high reactivity. Also they could act as nanofiller, and densifying the microstructure as well as reducing the porosity of concrete (Zhang and Li 2011). The experimental results show that the steel fiber ratios and curing age noticeably influenced the fracture properties of steel fiber reinforced concrete (Fu *et al.* 2014) and also it observed previously that all the fracture parameters are reliant on geometrical factor and specimen size (Kumar and Barai

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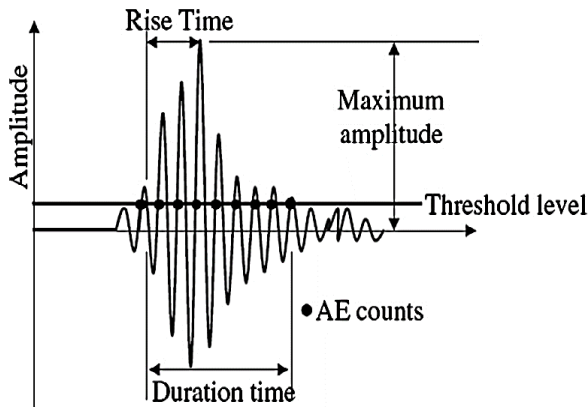


Fig. 1 Typical AE signal and parameters

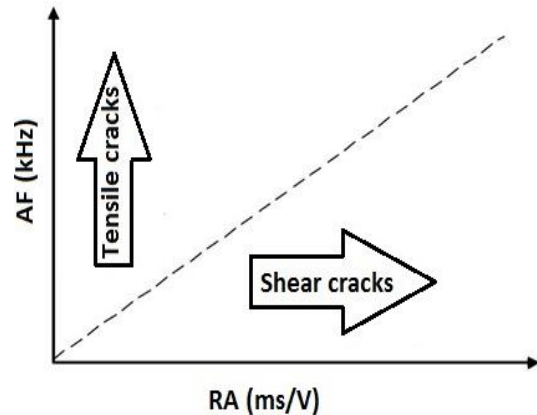


Fig. 2 Classification of crack type with arrangement of AF and RA values

2012).

Due to the importance of cementitious materials and also their fracture system, several researchers such as Zhang *et al.* (2013 and 2014c) and Elices *et al.* (2000) studied the performance of cement base materials and fracture mechanics of concrete. Furthermore, the effect of different material characteristics on properties of concrete or rock structures has been studied by acoustic emission technique. Aggelis *et al.* (2011) studied fracture process in fibre reinforced concrete. They showed that the average frequency shows a lower value when the main failure mechanism shifts from tensile micro-cracking, to macro-cracking with fibre pull out. Haneef *et al.* (2013) investigated Influence of fly ash and curing on cracking behavior of concrete and revealed that curing and fly ash replacement have intense effect on micro-cracking, stable crack and unstable crack propagation. Sabri *et al.* (2016) used rocks with particle size heterogeneity to consider fracture toughness and failure mechanism of rock samples.

## 2. Acoustic emission monitoring

Abrupt changes which take place locally in materials due to deformation, cracking, departure and so that cause of generated elastic waves all used for Acoustic Emission (AE) expression. Since AE is non-destructive (ND) assessment technique and very operational to monitor the beginning and the growth of cracks in both materials and structures, it has been widely applied for materials evaluation in laboratory (Minemura *et al.* 1998).

In compare to other ND approaches, however, AE is typically applied during loading, while most other methods are applied before or after loading of a structure or testing program. Conversely, AE is often used to detect a failure at a very early step, long before completely fails of entire material (Grosse and Ohtsu 2008).

Acoustic emission is more intensely dependent on the nonelastic deformations in a material. Consequently, only this technique is accomplished of detecting the formation of new cracks and the development of existing cracks or friction progressions (Grosse and Ohtsu 2008).

A typical AE signal with parameters of counts, amplitude, duration, rise time, threshold is shown in Fig. 1.

In the case of studying AE parameters, counts define as the number of sampling times that an AE signal surpasses a distinct threshold during one AE signal, amplitude, usually expressed in decibels, is the maximum value of the AE signal, duration states the time span from the starting point to the end point of the AE signal, and rise time is related to the time interval starting from the time of AE signal generation to the time of the signal achieving its maximum amplitude.

Two worthwhile parameters that obtained indirectly based upon AE signal parameters are termed as Raise Angle (RA) and Average Frequency (AF) which calculated from rise time divided by amplitude and count divided by duration respectively (Sabri *et al.* 2016, Grosse and Ohtsu 2008).

### 2.1 Study performance of cementitious material with AE approach

In addition, failure behavior take place with release of stored strain energy, nucleating cracks and generating elastic waves. Elastic waves due to crack nucleation are sensed as AE waves, which spread inside cementitious material and are detected by an AE sensor attached on the surface (Grosse and Ohtsu 2008).

Avoiding disastrous fracture is the ultimate aim of monitoring AE phenomena by correlating valuable information detected by AE signals with growing fracture progression or deterioration. In cement base materials, the classification of crack types is suggested, using the arrangement of AF and RA values. This classification method has been uniformed and is shown in Fig. 2 (Grosse and Ohtsu 2008).

Furthermore, parameters of AE method with the failure modes such as the damage development under bending test, and monitoring the damage of concrete structures in real time have been associated (Kurz *et al.* 2006, Schechinger and Vogel 2007, Aggelis *et al.* 2009, Fan *et al.* 2016).

Number of publications in many scientific research areas at the end of the last/beginning of this century devoted to nanotechnologies and nanomaterials, quickly increases. Meanwhile major results in the area were obtained by chemists and physicists, the common of new papers also

Table 1 Chemical and physical properties of Portland cement (wt. %) chemical properties

| Material | SiO <sub>2</sub> | Al <sub>2</sub> O <sub>3</sub> | Fe <sub>2</sub> O <sub>3</sub> | CaO   | MgO  | SO <sub>3</sub> | Na <sub>2</sub> O | K <sub>2</sub> O | Loss on ignition |
|----------|------------------|--------------------------------|--------------------------------|-------|------|-----------------|-------------------|------------------|------------------|
| Cement   | 21.89            | 5.3                            | 3.34                           | 53.27 | 6.45 | 3.67            | 0.18              | 0.98             | 3.21             |

Specific gravity: 1.7 g/cm<sup>3</sup>

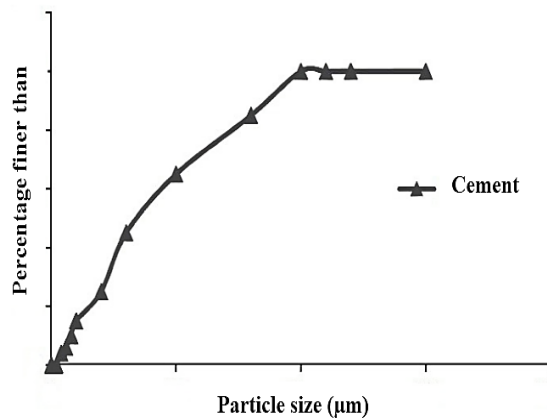


Fig. 3 The particle size distribution curves of OPC

come from chemistry, physics and materials science. In the same time, mechanics of materials such as failure and micro-crack growth behavior of this novel materials necessities further attention.

Despite of the previous and current efforts, only few studies are available, concerning the failure behavior of the mortar containing nano-silica particles.

Therefore, we conducted this investigational work and acoustic emission technique as an innovational and highly important work in the concrete and cement sciences.

The main purpose of this study is to consider the effects of the addition of the nano-silica particles to cement, on failure behavior by using valuable AE monitoring techniques. In this work efforts have been made to approve that it is possible to obtain high performance and high strength mortar by using nanomaterials that result in better mechanical and fracture properties.

### 3. Experimental program

#### 3.1 Cement

Ordinary Portland Cement (OPC) obtained from FiruzKuh Cement Manufacturing Company of Iran conforming to ASTM C150 (2003) standard and strength grade of 480 kg/cm<sup>2</sup> at the age of 28 days was used as received. The chemical and physical properties of the cement are shown in Table 1. The particle size distribution pattern of Portland cement has been illustrated in Fig. 3.

#### 3.2 Nano silica particles

Nano-silica particles with average particle size of 15 nm obtained from Aerosil Company of china were received and used. The properties of silica nanoparticles are shown in Table 2. The utilized silica nanoparticles due to their

Table 2 The general physical properties of silica nano particles

| Diameter (nm) | Surface volume ratio (m <sup>2</sup> /g) | Density (g/cm <sup>3</sup> ) | Purity (%) |
|---------------|--|------------------------------|------------|
| 15±3          | 160±12                                   | <0.14                        | >99.9      |

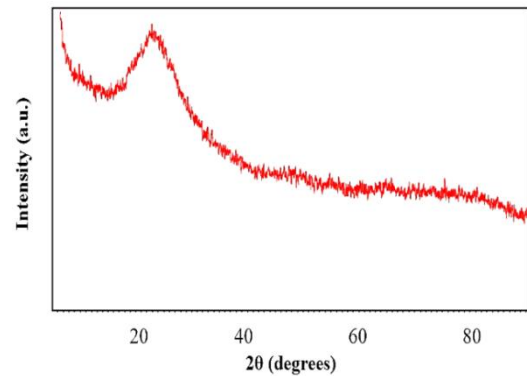


Fig. 4 XRD patterns of silica nanoparticles with average particle sizes of 15 nm

amorphous structure are of high pozzolanic reactivity and their spherical shape helps to enhance the physical properties of mortar. Moreover, the X-ray Diffraction (XRD) test indicates the amorphous structure of silica nanoparticles (illustrated in Fig. 4). As it can be seen, the XRD patterns show an approximately broad peak centered about  $2\theta \approx 22^\circ$  which demonstrates the amorphous structure of utilized silica nanoparticles (Wansom *et al.* 2009).

#### 3.3 Aggregates

Locally available natural sand with fineness modulus of 2.25 and specific gravity of 2.58 g/cm<sup>3</sup> was used as fine aggregate.

#### 3.4 Mixture proportioning

Two series of mixtures were prepared in the laboratory trials. Series P0 mixtures were prepared as control specimens. The control mixtures were made of natural aggregates, cement and water. Series N were prepared with different contents of nano-silica particles with average particle size of 15 nm. The mixtures were prepared with different amounts of nano-silica particles with 0.5%, 1%, and 1.5% by weight.

#### 3.5 Preparation of test specimens

Series N mixtures were made by mixing the fine aggregates and powder materials (cement and nano-silica particles) in a laboratory. For enhancing spreading of nano-silica particles, they were stirred with some of the mixing water at high speed (120 rpm) for 1 min and then added to the mixture (Jo *et al.* 2007). The powder material in the series P0 mixtures was only cement. They were mixed in dry condition for 2 min, and for another 3 min after adding the water. 50-mm Cube samples were molded in accordance with ASTM C109 (1993). The samples were covered with



Fig. 5 Servo-electric load frame and AE sensor

Table 3 Variation of compressive strength for both cement mortar specimens

| Sample tags | Nano-Silica particles (%) | Compressive strength (MPa) |        |         |
|-------------|---------------------------|----------------------------|--------|---------|
|             |                           | 3 days                     | 7 days | 28 days |
| P0          | 0                         | 6.86                       | 12.4   | 24.05   |
| N1          | 0.5                       | 9.02                       | 14.65  | 26.86   |
| N2          | 1                         | 11.6                       | 17.95  | 31.7    |
| N3          | 1.5                       | 12.05                      | 18.5   | 27.17   |

polyethylene sheets and moistened for 24 h. Then the samples were demolded and cured in water at a room temperature prior to testing. The compressive strength tests of aforementioned samples were done after 3, 7 and 28 curing period days. Reported results are averages of three trials.

An experimental setup including a servo-electric testing machine with a data acquisition system and an AE monitoring system was employed. The sensor was attached to the surface side of the specimen, as it can be seen in Fig. 5, for fracture behavior monitoring of cubic specimens. Ultrasonic gel was consumed for acoustic coupling though the sensor was held by the use of electric mounting tape during the experiment. Signal data are digitized by taking on a data acquisition system. A threshold of 38 dB was used as well, to eliminate the surrounding noise (Nejati and Ghazvinian 2014).

## 4. Results and discussion

### 4.1 Compressive strength

The compressive strength results of both two series P0, N1, N2 and N3 mixtures with different period test age 3, 7 and 28 days are shown in Table 3.

Test results of hardened cement mortar exposed that in mixtures with nano-silica particles the compressive strength was enhanced with incorporation of nano-silica particles up to 1.5% for 3 and 7 test ages. On the other hand, maximum compressive strength of 28 curing days obtained from specimens with 1% combination of nano-silica particles. It is worth declaring that for samples of 28 curing period, strength increased up to maximum with addition of 1% of

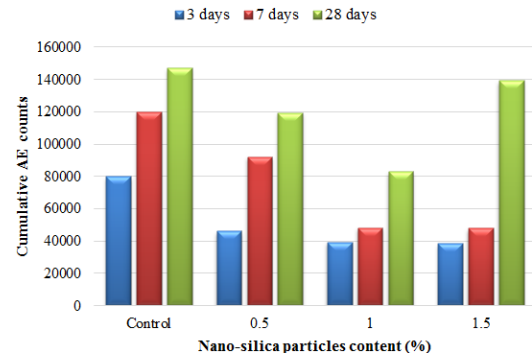


Fig. 6 Cumulative AE counts during compressive load for both types of cement mortar specimen at different age of tests

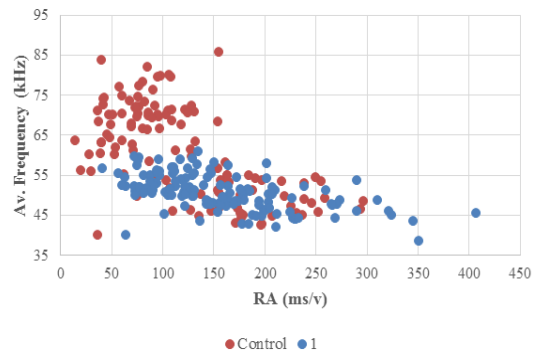


Fig. 7 RA value vs. average frequency correlation for control and nano-silica particles cement mortar specimens for curing of 28 days

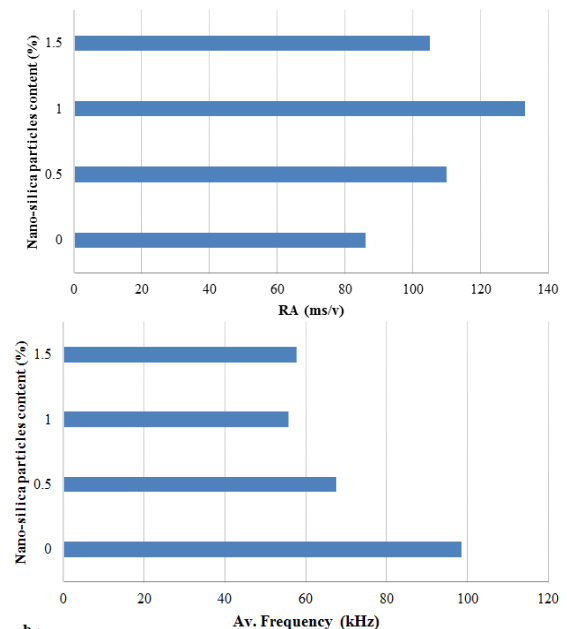


Fig. 8 Average (a) RA value, (b) AF value vs. nano-silica particles content for curing of 28 days

nano-silica particles to cement and then decreased which is near to the control specimen.

Decreasing of strength after adding quantity of nano-silica particles (1%) may be due to the fact that the amount of nano-silica particles present in the mixture (pozzolan) is

higher than the amount required to combine with the liberated lime during the process of hydration thus leading to surplus silica leaching out and cause of a deficiency in strength as it added to the cementitious material but does not contribute to compressive strengths (Al-Khalaf and Yousift 1984). Also this condition may be illustrated due to the fact that the surplus of nano-silica can alter the original amorphous C-S-H glue (lower Ca/Si ratio) into C<sub>5</sub>S<sub>6</sub>H<sub>5</sub> (tobermorite) which has lower strength properties and increase the amount of silicate polymerization (Purton 1993, Kuo *et al.* 2006, Raki *et al.* 2010). Furthermore, it can be due weak zones that occurs to the defect in spreading of nanoparticles (Najigivi *et al.* 2013).

#### 4.2 Observing of AE parameters

Variation of AE cumulative counts generated during the compressive testing of all period cured control and nano-silica cement mortar is shown in Fig. 6. It can be comprehended from Fig. 6 that the variation of cumulative AE counts with compressive loading of specimens with nano-silica particles reduced as compared to the control specimens for entirely ages of curing period. Moreover exploit reduces of generated AE cumulative counts incline to be linear for 3 and 7 days of curing and for 28 days of curing tend to be non-linear.

Source of AE activity during compressive load and foundation structure failure of material can be occurred by different causes during typical stress-strain curve such as closure of pre-existing cracks and development of micro cracks over weak boundaries of particles within the cement mortar matrix and finally unstable cracks open through the cement paste and aggregate macro crack pattern formation. Meanwhile samples reinforced with nano-silica particles have a denser binding paste matrix because of nanoparticles pozzolanic plus voids and pores filling effects and generated fewer counts compare to control samples that can be seen through Fig. 6.

Three categories of crack propagation modes are well known in fracture mechanics which are: mode I (tension), mode II (shear) and mode III (tearing). Failure mode investigation from RA value and average frequency of the AE signals during testing program reported formerly (Ohtsu and Tomoda 2008, Soulioti *et al.* 2009). High frequency signals resembles tensile crack behavior (mode I), however high RA values shows shear sort of crack (mode II).

Micro cracks changing behavior during compressive load and data acquisition of AE signals from both control and nano-silica particles cement mortar with the addition of 1% of nano-silica particles can be seen in Fig. 7. As well as the amount of RA value and AF of both cement mortar samples were provided in Fig. 8. Considering to Fig. 7 and Fig. 8 control specimens exhibit the lowest RA and the highest AF for all the AE counts recorded throughout the tests. The inclusion of nanoparticles, even in just 0.5% of the addition of nano-silica particles leads to change RA value and AF of control cement mortars compare to reinforced specimens. As it can be seen the highest RA and the lowest AF belongs to specimens with the addition of 1% of nano-silica particles. As shown in Fig. 7, comparing both specimens, addition of nano-silica particles may be leads to

more active micro cracks shear mode, further reducing tensile mode of micro cracks that closely associated to the increased compressive strength of specimens with nano-silica particles.

Likewise, during specimen preparation, 70% of hydration products provides C-S-H glue with about 10 nm average diameter reported formerly (Ferraris and Gaidis 1992). Therefore, nano-silica particles can thoroughly fill weak zones like voids and pores of cement mortar specimens to make solid matrix.

#### 5. Conclusions

Effect of nano-silica particles on the failure mechanism and fracturing behavior of cement mortar specimens have been considered in this study using acoustic emission technique. Results of experimental tests were listed as follow:

- Analyses of AE parameters indicated that the addition of nano-silica particles have a significant influence on the behavior of micro cracks and failure mechanism of cement mortars.
- In the 3 and 7 days of curing, the most compressive strength belongs to the specimens with 1.5% nano-silica particles; but when the curing time increases to 28 days, the specimens possess 1% nano-silica particles achieve the most compressive strength.
- Cumulative AE counts have a meaningful correlation with compressive strength of specimens; so that the specimen with most strength produces least Cumulative AE counts.
- Raise Angle (RA) and Average Frequency (AF) of the emitted signals from the specimens were influenced by the amount of nano-silica particles. The specimens with nanoparticles compared to the control concrete specimens possess higher RA and lower AF. Therefore, it can be concluded that addition of nano-silica particle change the mode of induced fractures in specimens, from tensile cracks to shear ones.
- The most value of RA and the least value of AF belongs the specimen with 1% nano-silica particles. It means that the specimen with 1% nano-silica particles compared to the other specimens involve some more shear induced fractures.

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