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Nondestructive tests for defections detection of nanoparticles in cement-based materials: A review

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Abstract. To date, nondestructive tests (NDT) applications and advances in detecting the dispersion and defections of the nano concrete (NC) materials fields are very limited. The current paper provides a review of the dispersion efficiency of nanomaterials in cement-based materials and how NDT can be efficiently used in detecting and visualizing the defections and dispersions of NC. The review identifies the characteristics of different types of nanoparticles used in NC. Nanomaterials influences on concrete characteristics and their dispersion degree are presented and discussed. The main aim of this article is to present and compare the common NDT that can be used for detecting and visualizing the defections and dispersions of different kinds of nanomaterials utilized in NC. The different microscopy and X-ray methods are explicitly reviewed and compared. Based on the collected data, it can be concluded that the fully detecting and visualizing of NC defections and dispersions have not been fully discovered and that needs further investigations. So, the distinction of this paper lies in defining NDT that can be employed for detecting and/or visualizing NC defections and dispersions.

Keywords: defect detection and visualization; dispersion; microscopy; nondestructive tests; pulse-ultrasonic; X-ray

1. Introduction

Nanoscale materials that used in concrete often made different properties from their bulk conventional concrete. Whereas, the surface-to-volume ratio of nanoscale material increase its creativity at the molecular level (Mourdikoudis *et al*. 2018, Ashok *et al*. 2017). However, the size of these materials making the preparing of concrete more complex and challenging (Chalangaran *et al*. 2020, Mansouri *et al*. 2020). The main nano concrete difficulties that have negative effects of its performance are defection and dispersion of nanoparticles. At the same time, nondestructive tests (NDT) are growing demand for more advanced, reliable, and robust inspection and testing methods for visualizing the nano concrete defections and dispersions.

The main defections in concrete are due to the existence of the voids and cracks of surface and subsurface of concrete. Hardened concrete comprises different categories of voids which are changing in volume during the hardening process (Farzampour 2017, 2020). A large number of air bubbles created by air-entraining agents or by poor compaction act as “pressure-relief reservoirs” in hardened concrete. In some cases, mico-Nano air bubbles were used to improve particular concrete properties. Different studies were evaluated the performance of fresh and hardened concrete that include microbubbles for improving some characteristics of concrete (Kim *et al*. 2020, Arefi *et al*. 2016). However, the concrete voids are assumed as a reason for the reduction of concrete strength, it is assumed that about 5.5% reduction in strength occurs on every 1% additional air entrainment (Dewar 2003). Zeng *et al*. (2020) summarized the reasons of voids creation as follows: in the fresh concrete, the voids are generated during the mixing process and a few amount of voids produced from the air dissolved in water. The voids contain air entrapped inside and a thin external liquid membrane packing. These properties are affected by the categories and arrangements of surfactants absorbed on it (Zeng *et al*. 2020). “The membrane, which has micropores containing water, is the air-liquid interface of bubble and is one of the important factors affecting interphase mass transfer” (Zeng *et al*. 2020). Powers (1954) pointed out that the air bubbles were mainly entrained by the vortex which is generated during the stirring process, and entrapped by the three-dimensional barrier formed via the fine aggregates which were colliding with each other. Although different methods have enabled and applied for determining the voids positions and volumes, there are still limited for using in nanoparticles, especially in concrete admixtures.

Meanwhile, cracking is the most common types of failure of conventional and nano concrete materials. Many factors can cause the concrete cracks, such as extreme temperature, volume change from shrinkage and external load. The concrete is a nonhomogeneous material and its fracture process is complex, accompanied by microcrack initiation, propagation, branching, and coalescence. Many

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researchers attempted to simulate and model concrete cracks, such as the cohesive zone model (CZM), lattice model, rigid body spring method (RBSM), discrete element method (DEM), and extended finite element method (XFEM). CZM has found a better model for simulating the nano-crack of concrete (Dong *et al*. 2018). NDTs were applied to estimate and detect cracks width and length of concrete materials. Here, an attempt to review best NDTs that can be used to visualize the defections, voids and cracks, characteristics in nano-concrete materials.

The dispersion of nanoparticles in nano concrete materials can be considered as the most important factor that should be studied carefully. The dispersion efficiency affects both fresh and hardened nanomaterial characteristics. Dispersion of CNTs (carbon nanotubes) and CNFs (carbon fibers) is one of the major factors that strongly influence the performance/efficiency of nano-particles in concrete materials. Because of its physical properties, these nanomaterials have a strong tendency to agglomerate. The difficulties of infiltration of agglomerates with matrices and their presence are the main source of potential defects in nanocomposites. The dispersion is a process of deagglomeration and subsequent distribution of nanomaterials. Dispersion can occur either due to abrupt splitting up of agglomerates into small fragments under high stress (rupture) or due to continuous detachment of small fragments at comparatively lower stress (erosion) (Parveen *et al*. 2013). Different methods have been used to achieve homogeneous dispersion of carbon nanomaterials in water and various polymers such as using solvents, surfactants, functionalization with acids, amines, fluorines, plasma, microwave and matrix moieties, noncovalent functionalization, using block polymers, wrapping conjugated polymers, and other techniques. On the other hand, the dispersion of nanoparticles in cement paste, mortar or concrete is commonly difficult to measure. Here, an attempt to find a suitable technique can be used to evaluate the nanoparticles dispersions in concrete materials.

This paper aims to provide an overview of the NDTs that can be used to detect and visualize the nano concrete defections and dispersions. In section 2, nanomaterial of concrete is presented and evaluated, in addition, the main reasons for dispersions and defections in nanoparticles of concrete are collected and discussed. Section 3 propose the main NDT of different types of microscopy and X-ray techniques which can be used to detect and visualize the nanoparticles defections and dispersions in concrete materials. In section 4, the conclusions and research needs are summarized.

2. Nanomaterials characteristics and influences in concrete

Nanotechnology attracted considerable attention in the construction field because of its unique characteristics as well as the high potential to improve the overall performance of cement composites. It can be considered as a promising concept in concrete technology due to their high chemical reactivity and excellent physical performance (Singh *et al*. 2013, Wu *et al*. 2017, Xiao *et al*. 2019, Su *et al*. 2017, Khayat *et al*. 2019). Nanoparticles with particle size below 100 nm are widely known to modify and improve various characteristics of cement composites including porosity, durability and mechanical properties (Korayem *et al*. 2017, Jindal and Sharma 2020, Reches 2018). They are very reactive and accelerate the reactions of cement with water due to the high surface area/volume (Jindal and Sharma 2020). Moreover, they can work as fillers to fill the pores and densify the microstructure (Du *et al*. 2019). Nanoparticles have various influences on fresh properties of cement-based materials as follow: fill the voids between cement particles, reduce the free water content due to its high absorption, and tend to agglomerate due to van der Waals forces of attraction (Khayat *et al*. 2019, Reches 2018). They can improve the packing density of the cement matrix and densifies the interfacial transition zone between the matrix and the aggregate. Nanosilica, for example, can act as a perfect filler due to its extremely small size and can fill the voids between particles of microsilica and cement and consequently improve the packing of fine particles (Ghafari *et al*. 2014, Norhasri *et al*. 2017). These influences can increase the viscosity of the fresh mixture and affect the rheology in different aspects (Khayat *et al*. 2019). In addition, their inclusion accelerates the hydration rate of cement components leading to increasing the viscosity and yield stress of the fresh mixture (Meng and Khayat 2018, Paul *et al*. 2018). On the other hand, their addition can compensate the retardation influence exhibited by superplasticizer, since they have a nucleation ability to accelerate cement hydration. Kawashima *et al* used $CaCO_3$ nanoparticles to compensate for the negative influence of fly ash on the hydration of cementitious materials (Kawashima *et al*. 2014). However, their effect on increasing the viscosity of the mixture leads to entrapping more air bubbles in fresh concrete and increase the porosity of hardened concrete (Yu *et al*. 2014).

2.1 Influence of nanomaterials on concrete serviceability

Nanoparticles can be implemented for improving the resistance of cement composites to cracking and to physical and chemical deteriorations. In addition, they are reported to reduce the total shrinkage of concrete (Yang *et al*. 2015, Puentes *et al*. 2014), however, for drying shrinkage, the conclusions in the literature are contradictory (Wu *et al*. 2017). Some researchers confirmed that graphene oxide, nanofibers and nano TiO_2 can decrease the drying shrinkage of concrete (Yang *et al*. 2015, Zhang *et al*. 2015, Lu *et al*. 2017, Nik and Bahari 2011). This influence is due to the role of the very fine particles in refining the pores, particularly capillary pores and compacting the microstructure, and thereby reduces the water loss and the crack propagation. However, Gao *et al*. stated that nanosilica addition can increase the drying shrinkage due to the high water absorption of nanoparticles (Behfarnia and Salemi 2013). On the other hand, the addition of nanoparticles improves the concrete resistance to freeze-thaw damage due to the more compacted and denser microstructure (Fan *et al*. 2015, 2016, Gonzalez *et al*. 2016, Salemi and Behfarnia 2013). They consume the portlandite and produce additional C-S-H, which improve both the paste strength and the transition zone characteristics (Salemi and Behfarnia 2013). As consequences, the pore structure is refined, and the average pore size is significantly decreased leading to reducing the amount of water available for freezing and thawing process (Salemi and Behfarnia 2013). Similar results about the influence of nanoparticles on the resistance of cement mortar to acid attack were reported by Fan *et al*. (2016) and Ji (2005), the high reactivity of nanomaterials associated with the high filling ability enhanced the resistance to acid attack. Nanosilica is reported to enhance the resistance of chloride diffusion and water permeability (Aly *et al*. 2012). The use of nanomaterials improves the resistance of concrete to alkali-silica reaction due to the pozzolanic reaction (Said *et al*. 2012). Moreover, the addition of nanosilica densifies the microstructure of concrete and consequently reduce the damage caused by sulfate attack. With low porosity and fine pore size distribution, the penetration rate of sulfate ions is significantly slow down (Singh *et al*. 2013, Tobón *et al*. 2015, Sikora *et al*. 2019). The performance of concrete under high-temperature exposure is reported to be improved by the addition of nanoparticles (Chu *et al*. 2017, El-Gamal *et al*. 2018, Heikal *et al*. 2015, Horszczaruk *et al*. 2017, Irshidat and Al-Saleh 2018, Lim and Mondal 2015, Wang 2017, Amin *et al*. 2015, Shaikh and Supit 2015). The residual compressive strength of concrete after exposure to high temperature up to 800 °C is increased by incorporation of the optimal dosage of nanomaterial. The use of nanomaterials helps to form additional C-S-H which improve the transition zone characteristics and reduce the crack propagation after high-temperature exposure (Sikora *et al*. 2019, Irshidat and Al-Saleh 2018). On the other hand, it was found that incorporation of nanomaterials reduce the capillary pores, chloride diffusion and permeability and therefore, the rate of corrosion of reinforcement is reduced significantly (Ghafari *et al*. 2015a). Nanomaterials have two different influences regarding reinforcement corrosion. The first is consuming the calcium hydroxide due to the pozzolanic reaction and consequently, reduce the alkalinity and the passive layer efficiency around the reinforcement. The other influence which is more significant is modifying the pore size distribution and reduce the transportation ability of the cement composites and therefore improve the resistance to reinforcement corrosion (Hamed *et al*. 2019).

Nanoparticles can be divided into two main groups: fiber-like such as carbon nanotube and pozzolanic nanoparticles such as nano-silica, nano clay etc… (Morsy *et al*. 2011). The first group is characterized by their shape with needle action which improves the matrix tensile strength and consequently reduce crack propagation and bridge cracks (Tobón *et al*. 2015, Mendoza and Sierra, Tobón 2014, Parveen *et al*. 2013, Siddique and Mehta 2014). The second group has pozzolanic reactivity which increases C-S-H concentration in the system and improves mechanical properties (Singh *et al*. 2013, Du *et al*. 2019, Mendoza *et al*. 2014, Parveen *et al*. 2013, Kong *et al*. 2012). The optimum dosage of nanomaterials depends mainly on its surface area as well as on the amount of binder in the mixture and water/binder ratio. In spite of the beneficial roles of nanoparticles on technical properties of cement-based materials, their addition may cause some deficiencies such as low workability in addition to the high possibility of agglomeration due to high surface area and high van der Walls forces (Du *et al*. 2019, Paul *et al*. 2018). With increasing the dosage of nanomaterials above the optimum content, negative effects on both fresh, low workability, and hardened properties, low strength are reported (Paul *et al*. 2018) due to improper dispersion of nanoparticles (Bai *et al*. 2018). Dispersion of nanomaterial is the main factor that governs their successful applications in cement-based materials (Du *et al*. 2019).

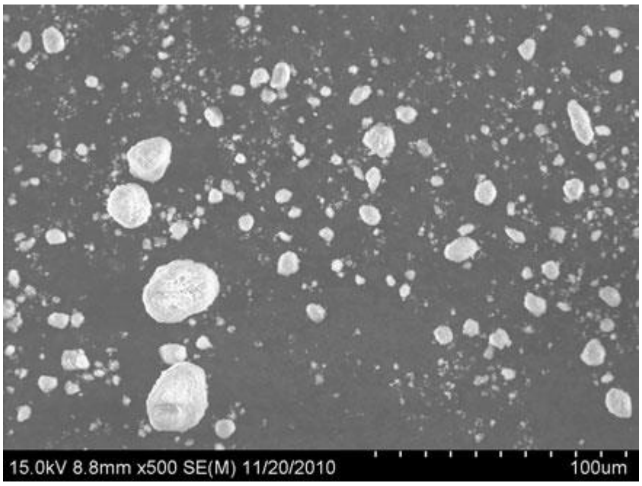


Fig. 1 SEM micrograph of agglomerated particles of powdered nano calcium carbonates (Kawashima *et al*. 2014)

2.2 Nanomaterials dispersion

With the development of nanotechnology, the superior properties of nanomaterials such as high specific surface area coupled with tiny size improved the characteristics of cement composites (Reches 2018). Effective dispersion of nanoparticles can significantly improve the fresh and hardened properties of cement composites (Reches 2018). Agglomeration occurred easily in nanomaterials system due to the high van der Walls forces resulting in defects and cracking in the cement matrix and hence low performance of concrete (Reches 2018) as can be seen in Fig. 1. The agglomeration of nanoparticles occurred mainly due to physical and chemical reasons. Van der walls forces of attraction and high surface area, small size of the particles are the main physical reasons for agglomeration. However, chemical reasons include the high reactivity of the nanoparticles as well as low pH which results in rapid agglomeration of particles and inefficient dispersion. It was reported that about 99% of the surface area of nanomaterials is deactivated due to agglomeration (Meng and Khayat 2018). High agglomeration takes place with all nanoparticles in the size range of 1 – 100 µm (Korayem *et al*. 2017, Paul 2018, Reches 2018). Generally, different types of nanoparticles agglomerate in aqueous medium such as cementitious pore solution. It happens due to the large number of very tiny nanoparticles owing to the very high surface energy (Gutierrez *et al* 2019). The agglomeration degree of nano SiO_2, nano TiO_2, nano Al_2O_3, nano Fe_2O_3 and nanoclay depends on pH, characteristics of particles and surface charge (Reches *et al*., 2018). The lack of an efficient method of dispersion constraints the application of nanoparticles in the construction field (Meng and Khayat 2018).

2.2.1 Dispersion methods of nanoparticles

Several methods have been developed to achieve homogeneous dispersion of nanoparticles in water such as using surfactants, solvents, functionalization with acids fluorines, microwaves, amines, plasma, matrix moieties, etc. Moreover, some physical techniques have been developed for that purpose such as ultrasonication which can be combined with the abovementioned chemical methods (Pérez-Nicolás *et al*. 2018). Superplasticizer was widely implemented as a dispersing material for nanoparticles in cement composites (Senff *et al*. 2009, Qian and Schutter 2018, Yu and Kwon 2009, Meng *et al*. 2019). It is found that PCE superplasticizer is a very effective method to disperse nanoparticles in the aqueous solution with a dosage of up to 2.5 %. However, with increasing the dose of nanoparticles to 5% the strength retrogression is observed due to agglomeration of nanoparticles (Kawashima *et al*. 2014). The size of the nanoparticles has a significant role in their dispersion and affects concrete properties significantly (Korayem *et al*. 2017). As the size of the nanoparticles decreases with a high surface area the tendency to agglomerate is increased with poor dispersion and the performance of concrete is decreased (Lavergne *et al*. 2019, Yazdanbakhsh *et al*. 2010). Moreover, it is reported that by increasing pH, zeta potential is reduced and consequently agglomeration occurred (Morsy *et al*. 2011, Chuah *et al*. 2018). To solve these clumping problems an effective method of dispersion should be applied (Reches *et al*. 2018, Yazdanbakhsh *et al*. 2012). The large difference between the size of nanoparticles and cement grains lead to poor dispersion of the system (Bentz *et al*. 1999). A method to improve the dispersion is by reducing cement particle sizes using ball milling but on the other hand, very small cement particles have other disadvantages such as high shrinkage, thermal cracking and high water demand (Collins *et al*. 2012). It should be noted that using chemical methods alone will not lead to a homogeneous distribution of nanoparticles, but it can improve the dispersion stability. Parveen *et al*. reported that the homogeneous distribution of nanomaterials in water does not mean a good dispersion in the nanocomposites due to the influence of different ions in pore solution (Reches 2018, Parveen *et al*. 2013). The use of chemical dispersants is necessary to improve the dispersion stability. The use of surfactants improves nanomaterials dispersion by minimizing water surface tension leading to stable dispersion. Therefore, it is recommended to combine both chemical and physical dispersion methods. Some types of concrete admixtures such as polycarboxylate (superplasticizer) can be used as an effective dispersant of nanomaterials (Feng *et al*. 2020). Feng *et al*. (2020) studied the influence of nanosilica dispersion on the early age hydration of cement paste. Gesoglu *et al*. (2016) developed a shell core structure made of PCE superplasticizer and nanosilica to improve the dispersion of the particles. The developed materials are well dispersed for long period in the pore solution. It has been reported that for each nanomaterial there is an appropriate method and for each method, there are specific terms and conditions to achieve the optimum dispersion (Korayem *et al*. 2017). A proper dispersion can be achieved in two steps, the first is breaking the agglomerated particles using physical or mechanical methods. However, the second is the use of chemicals to stabilize the dispersed solution and prevent it from reagglomeration. Several researchers compared the influence of microsilica and nanosilica on the resistance of cement-based materials to sulfate attack (Arel and Thomas 2017, Atahan and Dikme 2011, Sanchez and Ince 2009).

2.2.2 Dispersion efficiency

Nanomaterials have a high tendency to agglomerate and aggregate around each other due to van der Waals forces and therefore affect concrete characteristics negatively. The inappropriate dispersion of nanoparticles is the main issue hinders their wide application in cement composites. The poor dispersion of nanoparticles in the matrix makes agglomeration, bundle, clusters, flocs which initiate defects in the matrix and consequently, reduce the mechanical properties of cement-based materials and increase stress concentration (Korayem *et al*. 2017, Senff *et al*. 2009, Chuah *et al*. 2018). Dispersion efficiency depends mainly on the concentration in the solution as well as on the type and structure of the surfactant. Indeed, there is no widely accepted method to quantify the dispersion grade of nanoparticles in the cement matrix. Indirect methods are used such as mechanical properties to reflect the agglomeration existence of nanomaterials. The quality of dispersion is evaluated according to the composite performance: uniform dispersion is reported when improved characteristics are achieved (Korayem *et al*. 2017). Abbas found that with a high content of nanosilica, negative influences on workability are occurred due to conglomeration and dispersion problems of particles (Abbas 2009). Jabri *et al* observed a decrease in compressive strength with increasing the nanoparticles content and revealed that to the high possibility of poor dispersion and agglomeration of nanoparticles which generate weak zones in the pore solution (Al-Jabri and Shoukry 2014). Homogeneous distribution of nanoparticles resulted in strong improvement in mechanical properties and reduction in shrinkage of cement composites (Yazdanbakhsh *et al*. 2012, Ghafari *et al*. 2015b). The dispersion efficiency depends on the particle shape, type and method of preparation (Mendoza *et al*. 2013). Hamed *et al*. studied the influence of sonication of nano clay on concrete properties (Hamed *et al*. 2019). It is found that bond strength, split tensile strength and compressive strength of concrete are significantly improved with the addition of nano clay due to its pozzolanic reactivity, needle effect, nucleation effect and filling effect which leads to well compacted and denser microstructure. All these effects strongly depend on the well

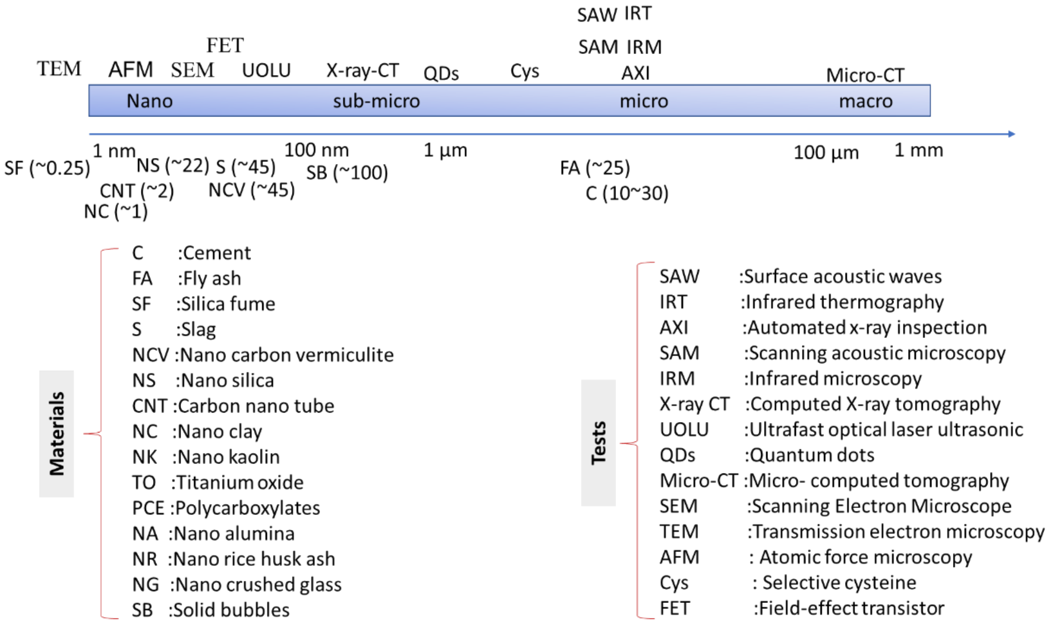


Fig. 2 Nanomaterials for concrete and tests (Fig. is reproduced from (Aryan *et al*. 2018))

dispersion of the nanoparticles. For homogeneous dispersion of nanoparticles within the aqueous solution, a certain amount of energy is required to break down agglomerated particles to their original sizes (Korayem *et al*. 2017). Thereafter, stabilization of dispersed particles is important in order to improve their efficiency and hinder their tendency to re-agglomerate (Korayem *et al*. 2017).

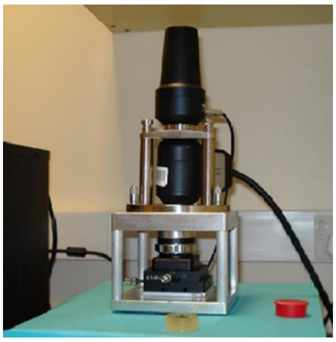
Cement hydration is accelerated and microstructure is improved depending on the shell/core ratio. Gesoglu compared the effect of nanosilica and microsilica on the performance of UHPC with low binder content (Oertel *et al*. 2014). The obtained results showed that 1% of nanosilica has similar performance as 10% microsilica. In addition, the best performance was achieved by combining both nanosilica and microsilica. A similar conclusion was observed by Sobolev *et al*. (2008), who attributed that to the better packing of the cementitious system. The distribution efficiency of any materials depends mainly on the nanoparticles properties as well as the matrix viscosity. In addition, homogeneously dispersed nanoparticles work as seeding for accelerating cement hydration (Paul *et al*. 2018, Sharma *et al*. 2019). Ghafoori *et al*. (2016) studied the influence of nanosilica on cement paste characteristics and compare its performance with that of microsilica. The flow spread of the paste is reduced by the addition of nanosilica due to the high water demand of the fine particles with a high surface area. It is found also that significant reduction in portlandite amount as revealed by TGA is associated with the addition of nanosilica. It is concluded that the main parameter control the performance of nanosilica is its dispersion in the matrix, when the nanoparticles are homogeneously distributed, the produced concrete has better resistance to sulfate attack than silica fume concrete. It is also reported that the addition of silica fume can work as wedges to separate nanoparticles mechanically (Li *et al*. 2016, 2020). It has a clear role in improving the dispersion and stabilization of nanoparticles. Bai *et al*. (2018) studied the influence of silica fume on the dispersion of graphene and found that the interfacial strength between graphene and hydration products is improved and the dispersion of graphene became more homogeneous with the addition of silica fume.

3. Nondestructive tests for detecting nano-concrete defections and dispersions

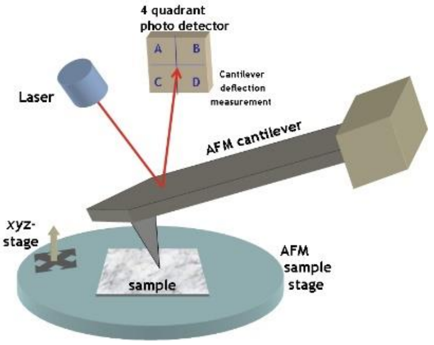
In this section, NDT that applied to estimate the defections and dispersions of different kind of materials including nano concrete were collected and discussed. From our state-of-the-art, different detection methods can be used to classify the nano concrete defections and dispersions. First, in the data processing, this category can be divided into signal and image processing to detect/visualize the nanoparticles of materials. Second, the processing of the machine used, this category can be divided into optics, laser, acoustics, magnetic, and pulse methods. Third, the estimating processing, and in this section, the category can be divided into processing during the measurement and processing after the measurement (predictive method or post-processing methods). The current study aims to provide a review of NDT for using these categories in nano concrete materials and the capability of using these tests in estimating the characteristics of materials. Fig. 2 shows the materials that may use to produce a nanomaterial and the common tests that used to estimate the material properties. Herein, one of common NDT methods that used in concrete defects detection is the electrical resistance testing (ERT) (Sanchez and Sobolev 2010, Nsengiyumva *et al*. 2021). Also, the piezoresistive sensors have been used to estimate the concrete defections (Zhang *et al*. 2016, Dong *et al*. 2019a, 2019b). The mechanical properties of concrete can

Table 1 Methods characteristics for surface and material sensitivity estimations (Khan *et al*. 2016, Eaton *et al*. 2017, Dufrêne *et al*. 2017, Philip *et al*. 2014)

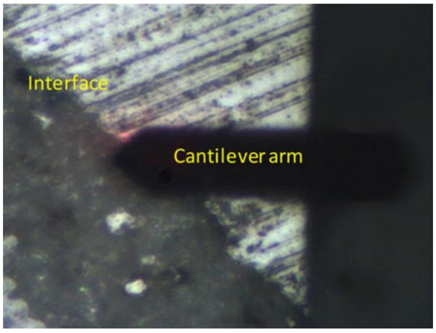
Parameter	SEM	TEM	AFM
Resolution (direction)	1 nm (XY)	0.1 nm (XY)	1 nm (XY), 0.1 nm (Z)
Physical basis	Emission of electrons	Scattering of electrons	Physical interaction with sample
Environment	Vacuum	Vacuum	Vacuum/air/liquid/gas
Material sensitivity	Somewhat increases with atomic number	Increases with atomic number	Equal for all materials
Surface height	Not possible	Not possible	Possible
Artefacts	Tip, force, scanning	Dehydration, ice crystal formation, beam damage	Dehydration, metal shadowing, beam damage
Cost	Expensive	Expensive	Cheap
Measurement speed	Fast	Fast	Slow



(a) (Khan *et al*. 2016)



(b) (AFM 2020)



(c) (Bisht *et al*. 2019)

Fig. 3 AFM machine (a), principal components (b) and tapping mode of AFM cantilever (c)

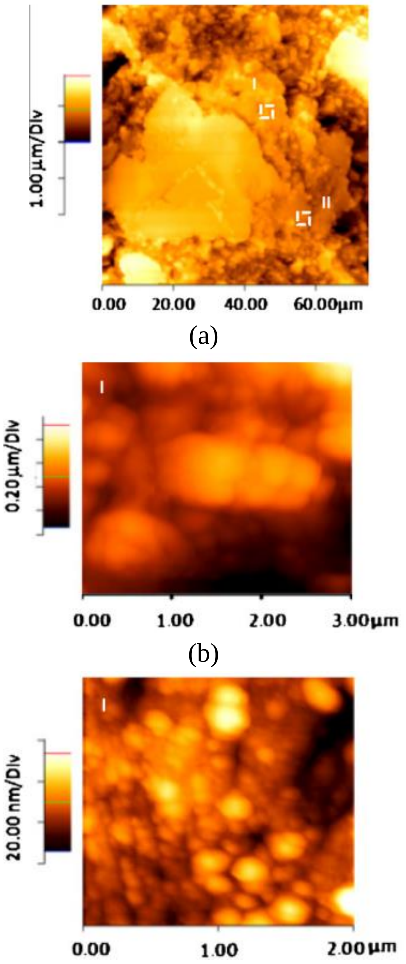
be measured using ERT (Mahdikhani *et al*. 2018). Concrete-based sensor was developed for real time monitoring of material behaviors (Dong *et al*. 2020a, 2020b, Ahmed *et al*. 2020, Guo *et al*. 2021). However, there are some disadvantages in this technique, that are the central filed of sample is measured, no bridge between the poles should be considered, the calibration should be over a very small range, and the vibration and heat make it harder to find flaws (Sophocleous 2017, Nsengiyumva *et al*. 2021). Moreover, although these methods have been used in detecting the defections of nano concrete, it is still limited for estimating the dispersions of nano particles. These methods operation principles, used and limitation are reviewed in (Rana *et al*. 2016, Cosoli *et al*. 2020, Nauman 2021, Nsengiyumva *et al*. 2021) and interested readers are directed to these review studies for more information.

As presented in Fig. 2, the TEM, AFM, SEM, UOLU, X-ray-CT, FET and QDs can be used to estimate the properties of the nanomaterials. These tests can be divided into microscopy, pulse-ultrasonic, X-ray, electric-pulse, and semiconductor methods. From the Fig., it can be seen that the microscopy tests have high accurate measurements to detect the nanoparticles characteristics. However, the X-ray techniques are commonly use in nanoparticles visualization for dispersion nanoparticles. Therefore, this review focused on different microscopy and X-ray methods. These methods are found more powerful for visualizing and detecting nanoparticles.

3.1 Atomic force microscopy (AFM)

The principal of AFM is a developed NDT technique utilized for estimating the material characteristics, such as morphology, size, surface roughness and texture, from sub-micron to nanometer length scale through local image processioning (Nasrollahzadeh *et al*. 2019, Khan *et al*. 2016). It is a very high-resolution of scanning probe microscopy (SPM) (Nasrollahzadeh *et al*. 2019). It is an efficient analytical test for nanoparticles which can be used in different environments, such as air, liquid or vacuum (Khan *et al*. 2016). The main advantages of AFM for investigating nanoparticles comparing to optical and electron microscopes techniques are the accuracy, cost, nondestructively and sample visualization in three directions, as well as the possibility to use for detecting and visualizing subsurface defections (Khan *et al*. 2016, Eaton *et al*. 2017, Shekhawat *et al*. 2017, Yip *et al*. 2019). In addition, it does not need any surface coating before scanning (Mourdikoudis *et al*. 2018). Table 1 presents a comparison between AFM, SEM and TEM.

AFM contains three important parts that are a cantilever for moving over the sample surface, laser source for generating a laser beam to the cantilever and photodetector to collect the data. Fig. 3 shows the diagram of AFM components and machine. The force that generated from the cantilever tip movement over the specimen is used to detect the surface characteristics through the evaluation of that oscillation frequency, phase and amplitude. Thus, the estimated frequency, amplitude and phase modes can be used for defining the martial characteristics. Here, there are

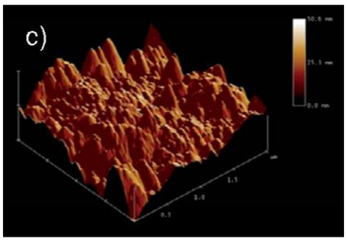


(a)

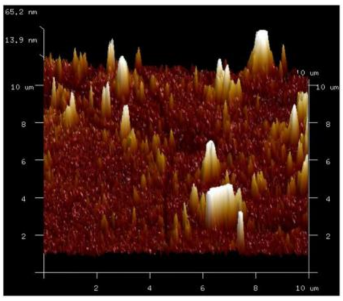
(b)

(c)

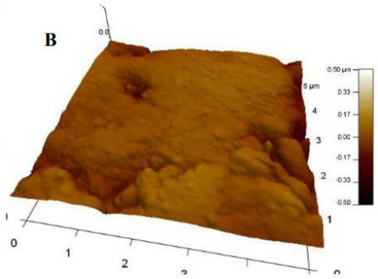
Fig. 4 The calcium hydroxide crystals (CH) phase of cement (a) sample and at two different regions (b) I and (c) II (Peled *et al.* 2013)



(a) (Sáez de Ibarra *et al.* 2006)



(b) (Wu *et al.* 2020)



(c) (Barreto and Brandao 2014)

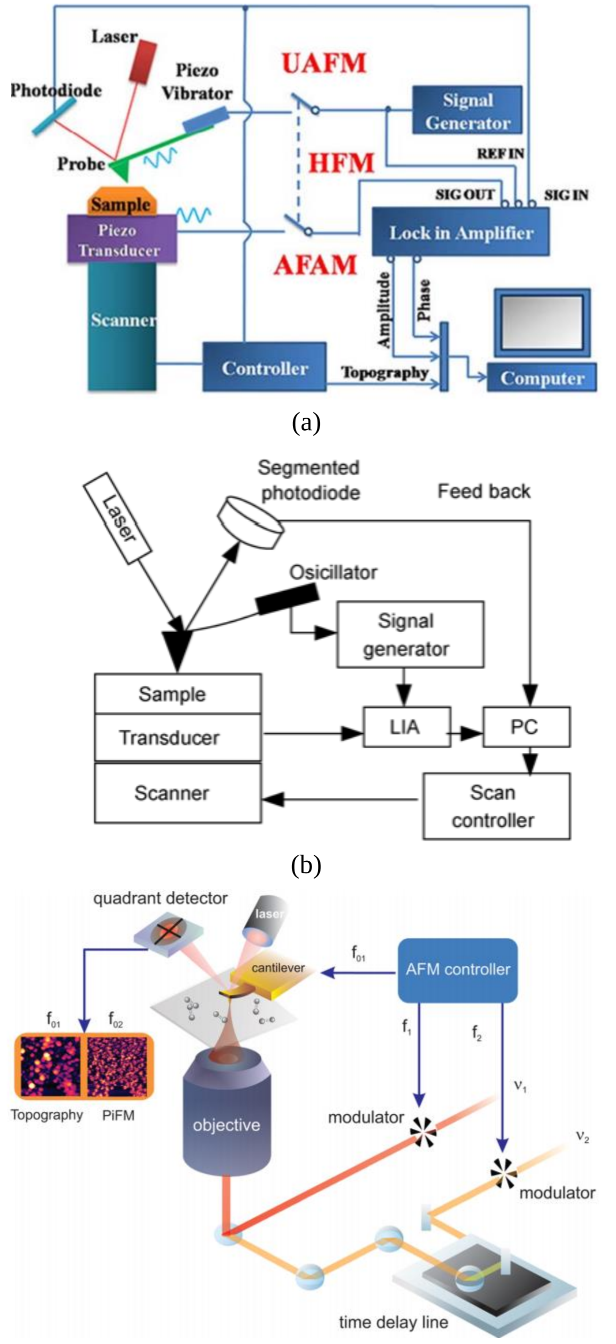
Fig. 5 3D topographic maps of concrete interaction with (a) CNT, (b) glasses and (c) ceramic

three techniques for using cantilever tip for surface detection, that are contact, non-contact and tapping modes (see Fig. 3(c)), the model's techniques, advantages and disadvantages were summarized in (Marrese *et al.* 2017). AFM can provide different models for different investigations, electron tuning spectra can be served for chemical identifications (Sugimoto *et al.* 2007), ultrasonic scattering can be used for subsurface detections (Shekhawat *et al.* 2017, Ahmed *et al.* 2020). Herein, there are many different variations of subsurface AFM imaging which can be classified as ultrasonic force microscopy (UFM), atomic force acoustic microscopy (AFAM), heterodyne force microscopy (HFM), scanning near-field thickness resonance acoustic microscopy (SNTRAM), and photoinduced force microscopy (PiFM) (Yip *et al.* 2019, Shekhawat *et al.* 2017, Jahng *et al.* 2015).

The AFM was used for estimating the mechanical and physical characteristics of concrete incorporating nanoparticles. For instance, AFM was found to be effective for determination of mechanical properties, Young's modulus, of nano-admixture of cement paste (Horszczaruk *et al.* 2020). Local elastic and damping properties of materials can be accurately estimated using AFM techniques (Phani *et al.* 2016). The interaction of cement particles with superplasticizer was evaluated in a different environment using AFM (Ferrari *et al.* 2010). Cement hydration, as presented in Fig. 4, can be also investigated and assessed using the AFM techniques (Peled *et al.* 2013). Different phases, such as calcium silicate hydrate (CSH), hydration cement particles and voids of cement mixture contains glasses were determined and detected by AFM (Bisht *et al.* 2019).

Three directions (3D) topographic profile can be estimated using AFM for evaluating the physical properties and surface defections. The microtopographic characterization by AFM for cement admiration proved the classical morphology of the important phases, such as ettringite, portlandite and calcite in the samples at the nanometer scale (Barreto and Brandao 2014). The reaction of Alkali-silica and carbon nano-tube (CNT) in concrete were investigated and evaluated using AFM (Wu *et al.* 2020, Sáez de Ibarra *et al.* 2006). Fig. 5 presents 3D topographic maps of CNT, glasses and ceramic reactions in concrete.

On the other hand, although the advantages of AFM for detecting and visualizing the surfaces defections and that capability for estimating characterize of nanoparticles, the application of AFM for detecting subsurface defections in nano-concrete is still limited. In concrete nanoparticles, the



(a)

(b)

(c)

Fig. 6 Diagram of the experimental setup of the (a) AFAM, UAFM, HFM and their derivates (Wang *et al*. 2017), (b) SNTRAM (Xu *et al*. 2011), and (c) PiFM (Jahng *et al*. 2015)

subsurface defections may find in a distance from the surface do not allow the force wavelength of AFM to reach them. “High constriction at higher frequencies in the sending medium restricts the infiltration profundity” (Shekhawat *et al*. 2017). AFM was developed to UFM by integrated with ultrasonic forces, and ultrasonic atomic force microscopy (UAFM) which is one of this kind on integration, also, AFAM and HFM use ultrasonic in the scanning, PiFM combine AFM with optical absorption spectroscopy, and SNTRAM integrate AFM with scanning near-field acoustic (Shekhawat *et al*. 2017, Yip *et al*. 2019, Broughton and Nunn 2006, Bertocci *et al*. 2019, Balke and Tselev 2018). Fig. 6 illustrates the diagram of experimental setups and layouts of the AFAM, UAFM, HFM, SNTRAM and PiFM.

However, the integrated techniques with AFM stage are still a creating region of examination, where understanding the material science of the component of identification and deciding the affectability of recognition of surface or subsurface properties are still in progress. A few studies were accomplished to detect subsurface defections of nanomaterials based on AFM techniques. Table 2 summarizes the concepts, advantages and limitations of different AFM techniques in NDT for defection detecting. But, in nanoscale materials of concrete application, there are no applications were conducted till now.

The surface and subsurface cracks of glass nanocomposite materials were visualized and detected using UAFM (McGuigan *et al*. 2002). Also, it was used to visualize the subsurface carbon position in graphite material (Tsuji and Yamanaka 2001). More applications and principals for using UAFM in visualizing and detecting subsurface defections can be found in (Yamanaka and Tsuji 2013, Shekhawat 2005, Piras *et al*. 2020, Stan and Solares 2014, Vitry 2016, Turner 2005, Yamanaka *et al*. 1999). The concept for using AFAM is the same for the UAFM in subsurface defection detection of nanomaterials, as presented in Table 2. AFAM was successfully applied for quantitative measurement of adhesion parameters of metals on the nanoscale (Hurley *et al*. 2006). In addition, holes under graphite were visualized using AFAM, see Fig. 7, (Yip *et al*. 2019). Amplitude and phase of AFAM signals can be used to visualize and detect the materials defections and material characteristics, such as cracks, steps and highly oriented pyrolytic graphite (HOPG), see Fig. 7.2 (Wang *et al*. 2017). More investigation for using AFAM for detecting of materials defections can be found in (Stan and Solares 2014, Vitry 2016, Hurley *et al*. 2005). Both techniques, UAFM and AFAM, can be integrated and used for visualizing three direction subsurface defections (Angeloni *et al*. 2018). And, HFM is one of that combination of UAFM and AFAM, as presented in Fig. 6, the detail of HFM and the comparison between it and other AFM techniques were concluded and presented in (Cuberes 2008). The subsurface changes of material contrast were visualized using HFM (Verbiest *et al*. 2017). Subsurface nanoparticles were also detected with a top-coat of the sample by 960 nm (Kimura *et al*. 2013). Furthermore, subsurface defections and characteristics by 500 nm were visualized and detected with a resolution at the 10- to 100-nanometer scale (Shekhawat 2005). Further investigation for using contact resonance AFM for estimating subsurface defections can be found in (Hurley *et al*. 2006, Angeloni *et al*. 2018, Reggente *et al*. 2017, Stan *et al*. 2009, 2012, Ma *et al*. 2017).

A near field AFM was found a good technique that can be used to estimate an accurate surface and subsurface defections and characteristics (Hirsekorn *et al*. 1996). Ultrasonic near-field optical microscopy, ultrasonic waves

Table 2 AFM techniques for NDT that can be used for visualizing/detecting nanoscale concrete defections

Technique	Principle	Advantages	Limitations	comment
AFAM	- sample stage excitation - probes deflection allows to visualize subsurface structures - resonance frequency shifts of AFM cantilever use to detect the surface and subsurface defections	- enables to image and measure local elasticity and damping of sample surfaces with a spatial resolution of the order of a few tens of nanometers. - AFAM phase is very sensitive to both contact stiffness and damping.	- tip wear and its influences should be understanding. - AFAM abilities to reveal sub 100 nm defects.	
UAFM	- cantilever base excitation - UAFM has modes of deflection and torsion. - probes deflection allows to visualize subsurface structures - resonance frequency shifts of AFM cantilever use to detect the surface and subsurface defections	- the images are very clear and sensitive to variation of material properties or to the existence of subsurface defects. - it is preferred for phase control detection applications. - the UAFM phase to contact damping can be used to track the resonance state by phase-control techniques	- work well with shallow depth samples (up to 0.2 μm) - if the excitation power is small, the resonance peak width decreases and the peak frequency increases.	The methods associated with atomic, acoustic, heterodyne forces are the most common to use for nanomaterials visualization and detection
HFM	- sample stage and/or cantilever base excitations - HFM exploits the soundwave that traveled through the sample. - HFM monitors the cantilever vibration at the beat frequency in amplitude and phase. - the phase cantilever response at the difference frequencies is expected to provide information about tip-sample interactions with increased time sensitivity.	- HFM can be used for delivering the highest contrast and resolution for subsurface detection. - HFM measurements penetrate the sample at most a few nanometers. - it can be used to defections below 82 nm thickness. - HFM possesses a high-frequency actuation for the time resolution inherent. - phase delays between tip and sample vibrations of the order of nanoseconds are easily detectable by HFM.	- the physical contrast mechanism is unknown	Using Phase-HFM, it can be used to distinguish the subsurface defects in contrast at identical thin polymer layers on the nanometer scale.
SNTRAM	- The piezoelectric transducer−sample assembly is mounted on a three-axis scanning translation stage in the AFM system. - A special silicon AFM cantilever is used to mount a second thickness mode piezoelectric transducer. - A portion of the photodetector output voltage is fed into a radio frequency lock-in amplifier.	- SNTRAM imaging technology has sharp phase contrast and mechanical sensitivity. - it provides a wide range of applications in nanomechanical imaging of semiconductor structures, as well as for other materials. - detected signals and images can be used to visualize material defections. - Subsurface features down to 5−8 nm lateral resolution have been demonstrated.	- the heating-induced changes to the cantilever's mechanical resonance - the image quality and scanning speed still need improving.	Subsurface defections can be visualized, and we think this method may be suitable in nano-concrete applications.
PiFM	- PiFM measures the photoinduced forces between a sharp tip and the sample. - it can consider the tip−sample junction to be as polarizable spheres and considering only dipolar contributions. - The light source in the PiFM system is a laser	- PiFM can be used to resolve the time-dependent excitation dynamics of chromophores. - PiFM can be conducted in the noncontact to soft-contact (tapping) mode. - a high spatial resolution can be given (10 nm or better) -it can be implemented in femtosecond illumination mode. - the PiFM signal is primarily from the surface (~30 nm)	- the heating-induced changes to the cantilever's mechanical resonance - PiFM places few constraints on the types of samples that can be studied	The sequential heterodyne configuration for PiFM can improve the accuracy in detecting surface and subsurface defections.

created by a pulsed laser and detected by a scanning near-field optical probe over a broad frequency bandwidth, was successfully used to detect the mechanical properties of a glass material under 200 nm thickness of silver (Ahn *et al*. 2013). Scanning near-field acoustic microscope possesses a high resolution, i.e. its lateral resolution approaches 10 nm (Xu *et al*. 2011). It was applied to estimate a subsurface defection in diamond-like carbon films that were 2–3 μm thick (Zinin *et al*. 2018). Here, the above methods used stress field that the tip generated at the surface to roughly estimated the depth range. Recently, SNTRAM has been developed to sensitively detect mechanical characteristics of

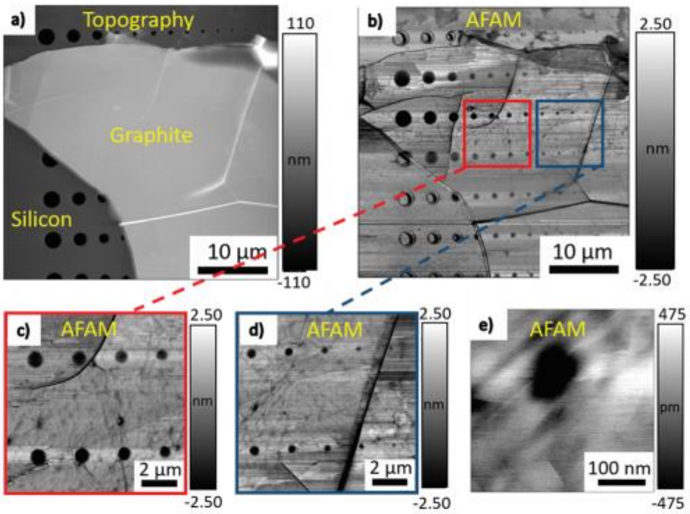
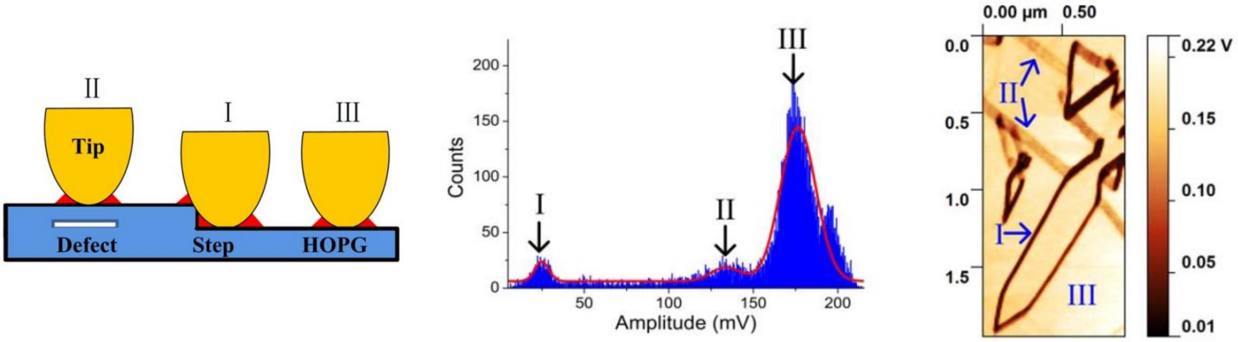
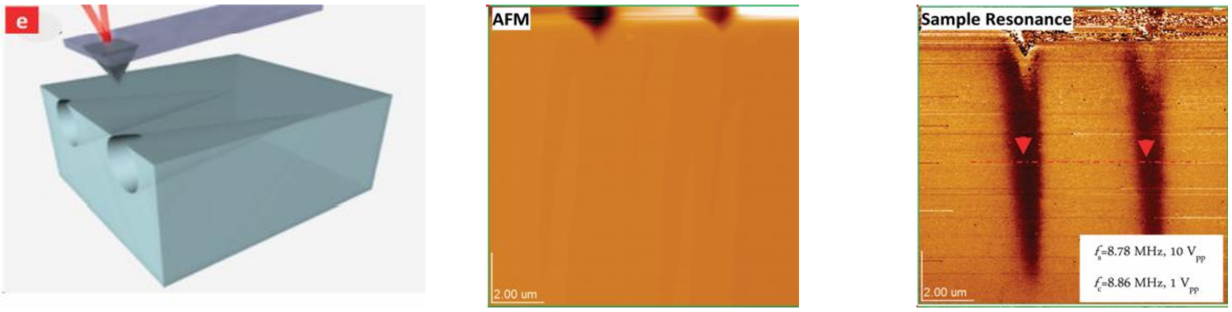


Fig. 7.1 Holes detected under graphite, (a) AFM topography of 80 nm thick graphite flake. (b) AFAM amplitude image at 325 kHz, 55 nN. (c) Holes ranging from 1–0.7 μm. (d) Holes ranging from 600–200 nm. (e) 50 nm hole with an apparent diameter of 100 nm. (Yip *et al*. 2019)



(a) (b) (c)

Fig. 7.2 AFAM imaging of the graphite. (a) Schematic illustration of the tip-sample contact mechanics in different regions, (a) Amplitude distribution among the entire scan area, (c) AFAM amplitude. (Wang *et al*. 2017)

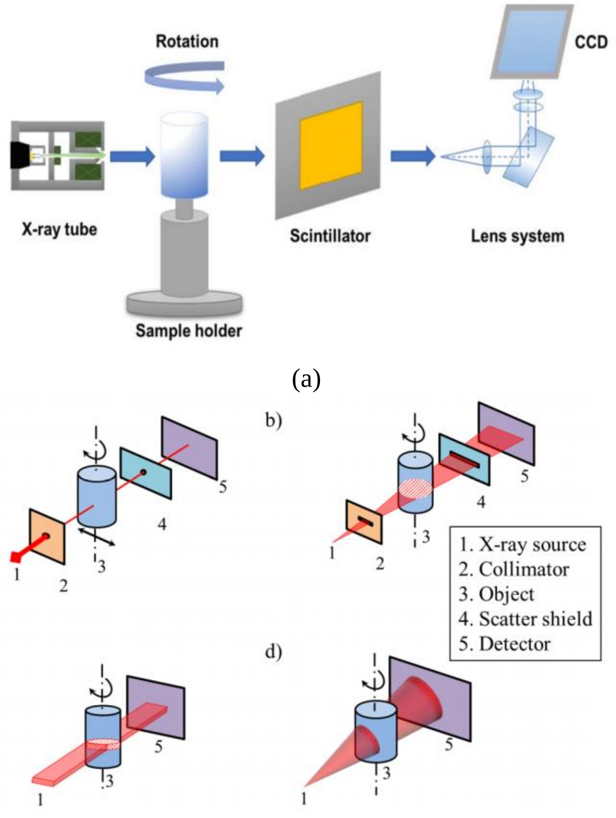


(a) (b) (c)

Fig. 8 holes detection of material, (a) sample diagram, (b) AFM image, (c) SNTRAM image (Shekhawat *et al*. 2017)

subsurface features (Shekhawat *et al*. 2017). Fig. 8 presents the difference between conventional AFM and SNTRAM for detecting the subsurface defections. From this Fig., it can be seen that it is a very promising method to estimate subsurface defections of nanoparticles in concrete applications. The main concept of SNTRAM is using the resonance of ultrasound holography signals for detecting the surface and subsurface defections. The ultrasonic waves created at the base of the example travel through the specimen bulk (Shekhawat *et al*. 2017). Diffraction of these waves by a subsurface element can be distinguished at the surface if the surface exists in the close to the field of this component (Shekhawat *et al*. 2017). Thus, it is expected that the AFM tip produces a stress field in the specimen. This stress field can get changed if there is a subsurface imperfection in its range (Shekhawat *et al*. 2017). The prevailing component of identification will rely upon the general sizes of the signs and sign irritations (Shekhawat *et al*. 2017).

More recently, the PiFM has been developed and used to



(a)

(b)

Fig. 9 Experimental concept of CT image (Jing and Yu 2017) (a) and X-ray and detector methods (b) (Eldessouki and Abdelkader 2019)

estimate more accurate subsurface mechanic properties of materials (Jahng *et al*. 2015, Wang *et al*. 2019, Fu *et al*. 2017). The design theory and applications of PiFM were concluded in (Jahng 2015). As it is a generally new technique, PiFM is not broadly utilized in the nano-concrete assessment. So, alongside imaging topography, PiFM gives mechanical imaging also. PiFM does this by estimating optical close fields in nanoscale structures. The spectroscopic data produced by PiFM has a spatiotemporal resolution of under 10 nm and broadband spectral sensitivity. Herein, although the heating due to the light source that generated between the tip and sample affect the mode change of the AFM cantilever, the PiFM accuracy is still high (Kim and Potma 2019). The results of previous studies show that PiFM can be used for building surface structure with nanometer-scale resolution (Fu *et al*. 2017). It can be integrated with infrared laser for estimating the materials defections and properties (Fu *et al*. 2017, Wang *et al*. 2019). The previous findings demonstrate the surface sensitivity of PiFM and highlight its potential to be employed as a complementary technique to confocal fluorescence microscopy (CFM) for understanding the catalytic properties of materials at different probing depths, analogous to the use of X-ray photoelectron spectroscopy (XPS) and energy-dispersive X-ray spectroscopy (EDS) to study the surface and bulk crystals, respectively (Fibikar *et al*. 2010, Fu *et al*. 2017, Almajhadi and Wickramasinghe 2017).

In conclusion, the AFM integrated techniques have been considered as promising methods to visualize and detect nanoparticles defections and dispersions of surface and subsurface in nano concrete applications with high resolution, especially using cantilever movement models to estimate materials characteristics. The AFM profile and spectrum are used to estimate the size and depth of nanomaterial defections (Jin *et al*. 2011, Kim *et al*. 2010, Ortega *et al*. 2016, Mondal *et al*. 2007). However, there is a compromise between the resolution and penetration depth of samples in metallic materials. The combination techniques attempt to overcome the drawback of subsurface defections. Here, the concrete characteristics may help to estimate the subsurface defections and dispersions based on specimen dimensions and used AFM technique. So, further investigation should be implemented in nano concrete to promote using AFM for visualizing that defection as an NDT in this field.

3.2 Computed tomography (CT)

X-ray CT imaging is an NDT for collecting a huge number of sector images for visualizing and detecting the internal microstructures of samples of interest (Hanke *et al*. 2016, Kim *et al*. 2012). The concepts of X-ray measurements, theory and applications can be found in (Hanke *et al*. 2016, Kim *et al*. 2012, Chae *et al*. 2013, Xavier *et al*. 2020). X-rays CT provides a promise resolution for defections detection in concrete structures (Chandrasekaran 2019). Herein, for the subsurface inspections, the hard X-ray is most suitable (Provis *et al*. 2011), while it can penetrate the sample depth more than the soft one. Fig. 9 presents the data collection concepts by CT and X-ray sources ((i) pencil, (ii) fan, (iii) parallel, (v) cone) designs for detecting or visualizing structures defections and dispersions. Sub-micron is the best resolution which was achieved by the parallel and cone beams of CT (Brisard *et al*. 2020). The main advantage of CT images is the high resolution of X-ray images with a rotation axis provides three dimensional (3D) view of specimens, which provides a good non-destructive test can be used to visualize the concrete characteristics and defections (Chae *et al*. 2013). As well as, 4D (3D plus changing with time) was detected and studied (Heenan *et al*. 2018). Moreover, the X-ray diffraction (XTD) uses for profiling the Crystal structure, composition, determine elemental composition and crystalline grain size (Mourdikoudis *et al*. 2018). Here, Scherrer equation used for estimating the latter parameter through the most intense peak of an XRD measurement for a specimen (Mourdikoudis *et al*. 2018).

Images processing steps for estimating CT 3D images are summarized in Fig. 10. X-ray CT scanning comprises three main steps: a beam of X-rays generates and sent through the material sample which set up on a rotating base, 2D digital images are collected through a flat-panel scintillator detector during each stage of rotation, then the software uses for reconstructing the 3D images (Bordelon and Roesler 2014). Meanwhile, the line profile parameters, as signal analysis, that extracted by XRD is a fast-on-line computer-controlled data that can be used as a quick

Table 3 Diffraction line-profile parameters for using in material characteristics (Balzar 1993)

Material characteristics				Method	identification
Position	Intensity	Shape	Shift		
√				Indexing	Cell parameter
√	√			Phase analysis	Identification and quantity
			√	Peak-shift analysis	Internal strain
√		√		Profile analysis	Microstrain, crystallite size, lattice defects
√	√	√		Structure refinement	Atomic positions, debye-waller factor

Table 4 Comparison of the CT techniques (Villa *et al*. 2019, Kutschera *et al*. 2011, Chae *et al*. 2013, Takeichi 2018, Jacobsen and Kirz 1998, Wargo *et al*. 2013)

	μ-CT	nCT	STXM	HPXRD
Main Features	images	images	images	Signal pattern
Time	~ 14 h	~ 35 h	3-72 Depend on the test phase	~70 ps
Image Resolution	500 nm	10* -60 nm	30-50 nm	--
Image Field of view	1-100 mm	~15 nm, at 50 nm spatial resolution	20-100 nm	--
General consideration	- soft or hard X-rays can be used - Vacuum not required	- soft or hard X-rays - Filtering process is required (Bossa *et al*. 2015) - Vacuum not required	- soft X-rays -a Fresnel zone plate) is often used to focus the soft X-rays. - Vacuum required	- The mechanical property and information of atomistic can be extracted - soft or hard X-rays - Vacuum not required

* with hard X-ray (Provis *et al*. 2011)

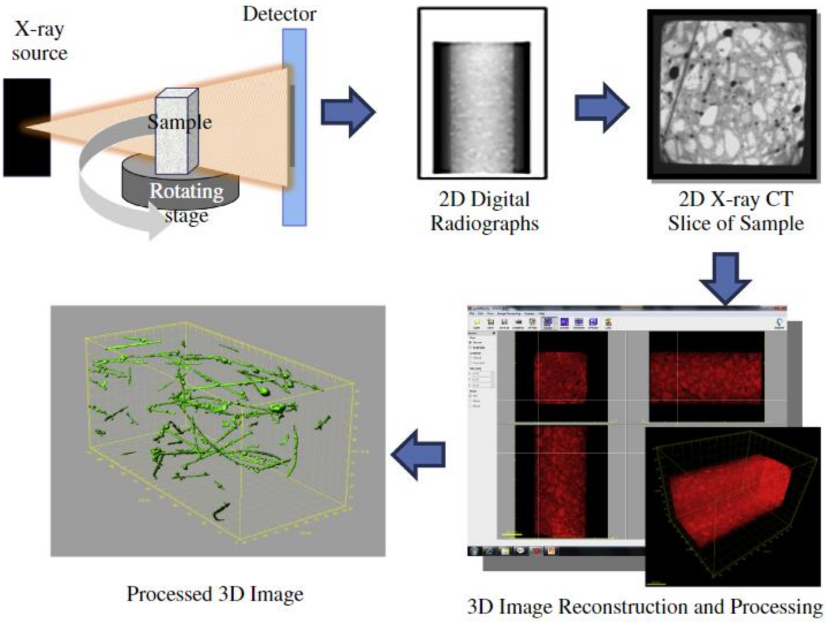
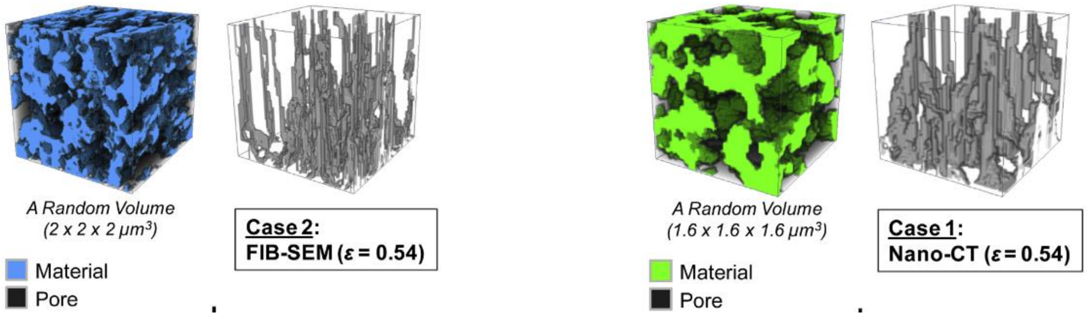


Fig. 10 Summary of image processing of CT images (Bordelon and Roesler 2014)

analysis of nanomaterial characteristics. This profile can be used to estimate the nanoparticles defections and dispersions. Table 3 proposes various types of using different diffraction line profile parameters.

Herein, the common X-ray CT techniques can be categorized into micro-CT (μ-CT), nanoscale CT (nCT), scanning transmission X-ray microscope (STXM), and high-pressure X-ray diffraction (HPXRD), that categorization is based on the image resolution, signal pattern detection and test purpose requirements. Table 4 illustrates the differences between these techniques. The most common type used for concrete inspections is the μ-CT technique. Surface and subsurface damages were visualized using in-situ 3D μ-CT images (Gonzalez *et al*.



(a) (b)

Fig. 11 Material and void distributions by (a) SEM and (b) nCT techniques (Wargo *et al*. 2013)

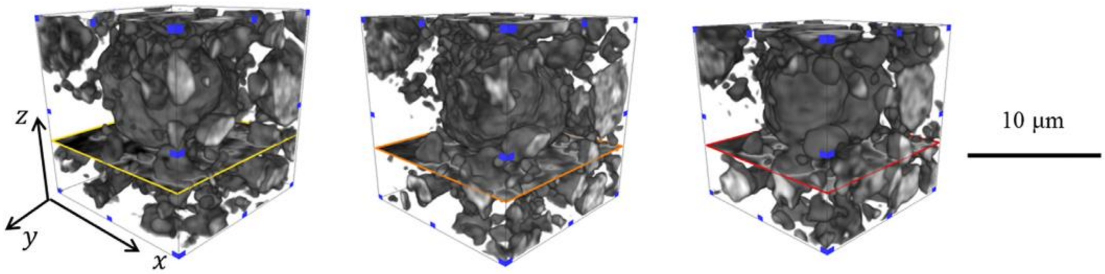


Fig. 12 Tracking the structural evolutions within a single particle from a lithium-ion battery electrode/current-collector sample thermally cycled in an air atmosphere: 3D surface render of a single large particle a) fresh, b) after the first cycle to°C200, c) after the second cycle to°C400 (Heenan *et al*. 2018)

2018). Also, the cement hydration was studied using X-ray techniques (Zhang *et al*. 2012, 2013). The summary of using μ-CT images in concrete and pavement materials was presented in (du Plessis and Boshoff 2019, Brisard *et al*. 2020, Gopalakrishnan *et al*. 2007). Nevertheless, nano-particles defections and dispersions may not be provided by enough resolution when using the μ-CT technique. Thus, nCT has been developed and used.

In general, many researches used CT for estimating concrete defections, voids and cracks, and dispersions. For instance, the voids of hardness concrete including carbon dioxide were visualized using 2D μ-CT, different regions of interest (ROI) was used to estimate the voids of the specimen, here the results showed that the concrete age affects the void ratio, the void of different ROI of the concrete section was not changed at 7 days, while it was totally differences at 28 days, furthermore, the accuracy of 2D μ-CT technique is increased with more curing of concrete (Bernardes *et al*. 2015). 3D images of μ-CT were successfully used to estimate the air-voids of hard concrete contents, also, the results showed that the 2D images can be misleading the air-voids information (Kim *et al*. 2012). Furthermore, 3D μ-CT was used to visualize the void distributions of lightweight aggregate, foamed concrete and cement past (Chung *et al*. 2013, Chung *et al*. 2015, Chung *et al*. 2016, 2017), the authors used the probability of phase information of the two-point correlation function to estimate the distribution. Also, by the same concepts, the void distribution and temperature effects were investigated for lightweight cement pastes containing fly ash and cement by 3D μ-CT (Abd Elrahman *et al*. 2020). More recent detail for using CT in visualizing the voids in concrete materials can be found in (Brisard *et al*. 2020, du Plessis and Boshoff 2019). Moreover, some researchers used nCT for visualizing concrete contents and characteristics. For example, nCT was used for detecting the distribution and localization of concrete contents (Provis *et al*. 2009). The voids of solid concrete that contain fly-ash were detected using nCT, and the results showed that it can provide a 3D view of nanoscale voids (Provis *et al*. 2011). Furthermore, 130 nm voids were detected by nCT, in addition, the combination of nCT and μ-CT improved the nanoparticles voids detections (Bossa *et al*. 2015). Here, a comparison was conducted between SEM and nCT for visualizing the porosity, pore connectivity, tortuosity, structural diffusivity coefficient, and chord length (i.e., void size) distribution of nanomaterials (micro-porous layer (MPL) specimen of Polymer electrolyte fuel cells with nano-scale pore feature), and the results showed that the nCT can provide a good resolution for estimating material's properties, see Fig. 11 (Wargo *et al*. 2013).

Recently, 4D images have been collected and evaluated by nCT technique with a field of view approach to 64 μm, voids and material changes were studied during the time curing, thermal effects on a solid concrete contains solid oxide fuel cells and lithium-ion batteries was investigated, see Fig. 12 (Heenan *et al*. 2018). Here, it is especially appropriate to 4D and in situ tries, where the same sample can be imaged on various occasions after some time or under changing conditions, permitting examining microstructural development. From these results, this technique can lead to an improved fundamental understanding of the defections and dispersions reasons and changes in nanoparticles of concrete materials.

Table 5 Summary of some recently available studies (2010~now) for using X-ray techniques in detecting the defections and dispersion of concrete materials

Small contents in admixture	Defection/dispersion	Type of material	CT technique	Ref.
Silica fume	Cracks, aggregate dispersion	Concrete	Medical CT	(Balázs *et al*. 2018)
Polymer macro-fiber	Air void, fiber dispersion	Concrete	μ-CT	(Bordelon and Roesler 2014)
cement	macrocracks	Cement paste	μ-CT	(Ren *et al*. 2015)
Aer solids	Air void	Cement paste	μ-CT	(Chung *et al*. 2016)
Fly ash	Cracks	Cement mortar	μ-CT	(Darma *et al*. 2013)
Alkali-resistant glass fiber	Air void	Concrete	μ-CT	(Lu 2017)
sodium lauryl ether sulfate	Air void	Concrete	μ-CT	(Kim *et al*. 2012)
Silica fume	Air void, fiber dispersion	Cement mortar	μ-CT	(Qsymah *et al*. 2017)
Cement	Air void, admixture content dispersion	Concrete	μ-CT	(Leite and Monteiro 2016)
Cement	Steel fiber dispersion	Cement mortar	μ-CT	(Mu *et al*. 2017)
Cement	Cracks in concrete and aggregate	Concrete	μ-CT	(Oesch *et al*. 2020)
CNT	Chemical reaction	Cement paste	XRD	(Ahmed *et al*. 2019, Jung *et al*. 2020)
Calcium sulfate whisker	Chemical reaction	Cement mortar	XRD	(Wan *et al*. 2019)
Tetracalcium aluminum carbonate	Chemical reaction	Cement pate	HPXRD	(Moon *et al*. 2012)
coal gangue	Sulfate erosion	Cement pate	XRD	(Qin *et al*. 2021)
Cement	Cracks and voids	Concrete	nCT	(Dong *et al*. 2018)
tricalcium silicate	voids	Cement pate	nCT	(Chen *et al*. 2019)
Cement	dispersion of steel fibers	Concrete	X-ray images	(Eva *et al*. 2020)
Fly ash	dispersion of steel fibers	Concrete	X-ray images	(Raju *et al*. 2019)
Nano silica	Nano-silica dispersion	Concrete	X-ray	(Ban *et al*. 2020)

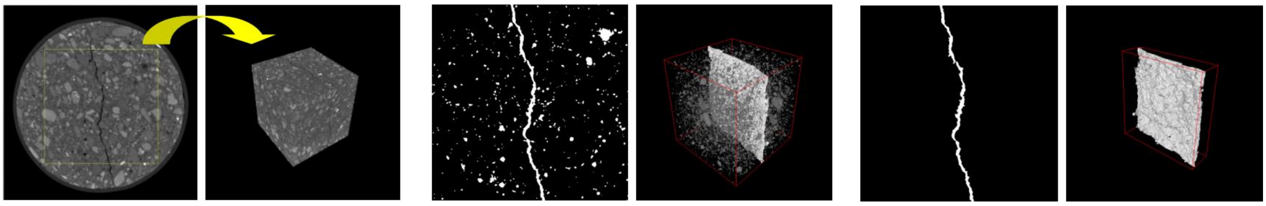

(a) (b) (c)

Fig. 13 Extraction and visualization of crack space from microtomographic images. (a) grayscale image and volume of interest (VOI), (b) Void segmentation of the VOI, the void and solid voxels are imaged as white and black, respectively, and 3D image of void space where the largest void cluster is imaged as white. The dead-end pore and isolated pore in the VOI are imaged as grey, (c) Crack space as the largest cluster is extracted, and 3D image of crack (Darma *et al*. 2013)

Furthermore, the potential of the CT techniques allows it to use as a good method for detecting macro cracks and those for characterizing micro/nano cracks (Naeimi *et al*. 2017). Table 5 presents recent studies for using X-ray techniques in estimating cracks in concrete materials. In general, cracks can happen through different factors, such as environmental effects (i.e., temperature changes), chemical factors, shrinkage and overloads. Microfocus X-ray CT was found a suitable technique to visualize nano-concrete as presented in Fig. 13 (Darma *et al*. 2013). Here, splits can be distinguished in CT pictures due to its undeniable quality of availability. CT has been a helpful apparatus to examine the spread of breaks. With the X-ray CT technique coupled with in-situ caesium tracer diffusion test, the crack would be further understood and visualized (Darma *et al*. 2013).

Meanwhile, the dispersions of nanoparticles in concrete materials should be investigated. Till now, little studies have been evaluated NDT methods that can be used to visualize the dispersions of nano concrete particles. Here, some of the X-ray tests that used in nanoparticles and concrete materials are proposed. The dispersion of gold (AuNP) with size 4 to 152 nm was studied and evaluated, the results showed that their X-ray attenuation linearly correlated with nano-particles dispersions (Dong *et al*. 2019c). Jung *et al*. (2020) used XRD to measure the CNT dispersion in concrete.

Table 6 Parameters needed to be determined for nano-particles defections and dispersions of nano concrete

Entity characterized	Characterization techniques suitable
Size (structural properties)	AFM
Shape	AFM, 3D-CT
Elemental-chemical composition	XRD
Crystal structure	XRD
Size distribution	AFM
3D visualization	AFM, 3D-CT
Dispersion of NP in matrices/supports	AFM

Table 7 Summary of the AFM and x-ray CT techniques that are used for nanoparticle characterization featured

Technique	Main information derived
AFM techniques	Nanoparticles (NP) size and shape in 3D mode, evaluate the degree of covering of a surface with NP morphology, dispersion of NPs in cells and other matrices/supports, precision in lateral dimensions of NPs, quick examination–elemental composition.
X-ray techniques (CT and XRD)	- Realistic 3D particle visualization, snapshots, video, quantitative information down to the atomic scale - Crystal structure, composition, crystalline grain size

The dispersion of fibers in ultra-high performance-fiber-reinforced concrete (UHPFRC) was visualized using X-ray CT (Zhou and Uchida 2017). Also, fiber dispersion was evaluated and detected using X-ray CT in the fiber reinforced concrete (Stähli *et al*. 2008). Further review articles for visualizing fibers and other concrete particles of a different kind of concrete have been presented in (du Plessis and Boshoff 2019). Thus, CT techniques can consider a very powerful tool for visualizing and detecting the internal distribution of nanoparticles in nano concrete. For more specific of nanomaterials in concrete structures, Table 5 demonstrates the most recent studies that used CT techniques for visualizing and detecting the materials defections and dispersions.

3.3 Summary and comparison of the proposed NDT techniques

Finally, In the cement-based materials, the pore structure is a critical factor that determines the internal morphologic organization. Hence the void/pore structure directly affects the key properties and performance of cement-based materials such as their impermeability, shrinkage, elastic modulus and strength. So far, the microstructure of cement paste has been widely studied in order to have a better understanding of the cement hydration process, and thus to develop better and more-dedicated cement-based materials. However, there are few studies on the internal pore structure of cement paste, especially in the nano-scale. This is partly because the cement paste is extremely complex and disordered, and partly because three-dimensional (3D) imaging techniques are needed for completing the measurements. Although it is hard to precisely measure the porosity of cement paste, there are 3 methods are usually used to estimate its quantity: gas adsorption, mercury intrusion porosimetry (MIP) and direct observation techniques including optical microscopy and AFM and x-ray CT. The material self-sensing has been used to estimate nano-concrete behaviors, however, Lee *et al*. (2017) and Zhang *et al*. (2019) showed that the amount of nano particles and water contents affects the sensitivity of these materials. Here, for best resolution, AFM may better to use for measuring the surface pores and x-ray CT may best for subsurface voids in different concrete processing stages, cement paste, mortar and fresh and hard concrete. 3D CT is better to visualize the pore percentage and evaluate the voids distributions. Furthermore, 4D CT can be used to study void positions and percentage during the curing time. In the same time, during concrete member service life, cracks arise in most of it due to various factors. Furthermore, it can affect concrete characteristics. Here, x-ray CT is a powerful tool that can be used to visualize the shape and size of cracks. The crack width can be presented in 3D by using CT images in the concrete section. The summary of NDT techniques that can be used in nano concrete defections and dispersions are presented in Tables 6 and 7.

4. Conclusions

The NDT of microscopy and x-ray techniques of nanoparticles in concrete has been reviewed and reported in the current research. Based on the collected materials obtained from previous studies, the following meaningful conclusions and research requirements can be drawn from this study:

- Homogeneous distribution of nanomaterials is the main parameter controlling its performance in cement-based materials. It depends strongly on the physical and chemical characteristics of the used nanomaterials as well as on the mixing method.
- Combination of both physical and chemical dispersion methods is recommended in order to break the bundles and clusters of nanoparticles to achieve homogeneous dispersion and at the same time prevent the fine particles from reagglomeration.
- The AFM integrated techniques have been confirmed promising to visualize and detect nanoparticles defections and dispersions of surface and subsurface in nano concrete applications with high resolution, especially using cantilever movement models to estimate materials characteristics. The AFM profile and spectrum can be applied to estimate the size and depth of nanomaterial defections. PiFM is found more suitable AFM technique can be used for building surface structure with nanometer-scale resolution. However, there is a compromise between the resolution and penetration depth of samples in metallic materials. The combination techniques attempt to overcome the drawback of subsurface defections.

- The micro-CT techniques have been found more powerful for detecting and visualizing nanoparticles defections in concrete. At the same time, the nano-CT has found more precisely to detect and visualize nano concrete defections. The difference between micro and nano CT's techniques are the filtration processing in both techniques. The nano CT technique is still limited in visualizing nano concrete defection. 3D and 4D images can be estimated for the nanoparticles in concrete by CT methods.

There are some knowledge gaps or remaining challenges that need to be addressed nanomaterials NDT in the construction practices.

- The concrete characteristics may help to estimate the subsurface defections and dispersions based on specimen dimensions and used AFM technique. So, further investigation should be implemented in nano concrete to promote using AFM for visualizing that defection as an NDT in this field. Furthermore, subsurface defection detecting by AFM should be improved and may image processing can improve the AFM images.
- CT techniques have found a good tool for detecting and visualizing nano concrete defections, voids and cracks, but it's still limited in visualizing the dispersion of nanoparticles in concrete materials. So, we think the visualization of nanoparticles of nano concrete material should be evaluated and need more discussion to understand the change distribution of nanoparticles in nano concrete.

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Conflict of interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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