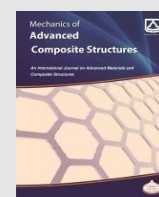




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## Research Article

# Effect of Microstructural Features and Al<sub>2</sub>O<sub>3</sub> Doping on Aging Resistance, Mechanical Properties and Crack Propagation in 3Y-TZP Plate Ceramics for Dental Restorations: A Comprehensive Peridynamics Study

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## ABSTRACT

This study investigates the effects of microstructural features, nanoporosity, and micro-cracks on macro-crack propagation in 3Y-TZP plate ceramics doped with Al<sub>2</sub>O<sub>3</sub> (0 to 0.5 wt%) for dental restoration applications, utilizing peridynamics (PD) theory for the first time. The research employs ordinary state-based PD to offer new insights into these interactions. Materials are synthesized through high-energy ball milling of ZrO<sub>2</sub> powders at 1500 °C for 2 h. Mechanical properties, including density, average porosity diameter, Young's modulus, Poisson's ratio, and fracture toughness ( $K_{IC}$ ), are rigorously assessed. Results indicate that increasing Al<sub>2</sub>O<sub>3</sub> content to 0.5 wt% enhances relative density, hardness, Young's modulus,  $K_{IC}$ , and flexural strength to 99.5%, 15.1 GPa, 280 GPa, 9.97 MPa·m<sup>1/2</sup>, and 355 MPa, respectively, while low-temperature degradation over 800 h shows that Al<sub>2</sub>O<sub>3</sub> doping significantly reduces aging kinetics. PD simulations demonstrate that micro-cracks substantially affect crack propagation, revealing a 15% reduction in macro-crack speeds compared to FEM results. This research enhances the understanding of dental ceramics and establishes a foundation for analyzing fractures in dental restoration ceramics using PD.

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## 1. Introduction

Zirconia (ZrO<sub>2</sub>) is a polycrystalline metal oxide ceramic known for its unique physical, chemical, mechanical, and clinical properties, making it suitable for hard biological tissues and dental restorations [1-2]. It is non-toxic and exhibits excellent bio-compatibility [3]. ZrO<sub>2</sub>'s high strength and fracture toughness ( $K_{IC}$ ) enhance its desirability in dentistry, where it

closely mimics the natural appearance of teeth [4].

To mitigate phase transformations that can cause micro-cracks, metal oxides like CaO [5], MgO [6], CeO<sub>2</sub> [7], Er<sub>2</sub>O<sub>3</sub> [8], and Y<sub>2</sub>O<sub>3</sub> [9] are added to pure ZrO<sub>2</sub>, resulting in partially stabilized zirconia (PSZ) that remains stable at room temperature [10]. PSZ can exist in cubic (c), tetragonal (t), and monoclinic (m) phases, with yttria (Y<sub>2</sub>O<sub>3</sub>) often used to favor the tetragonal phase [11]. Among these, 3 mol% yttria-

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tetragonal zirconia (3Y-TZP) is the most commonly used in dental restorations, including crowns, inlays, onlays, abutments, and implants [12]. Figure 1 illustrates some applications of 3Y-TZP in dental restoration.

Research has shown interest in doping 3Y-TZP with rare earth oxides. Zhang et al. [13] found that alumina ( $\text{Al}_2\text{O}_3$ ) co-doping enhances translucency and mechanical properties. Samodurova et al. [14] reported improvements in mechanical properties with  $\text{Al}_2\text{O}_3$  and silica co-doping. Seesala et al. [15] demonstrated that  $\text{Al}_2\text{O}_3$  coatings on zirconia implants improve strength and aging resistance. Salma [16]

explored microstructure development in YSZ-added  $\text{Al}_2\text{O}_3$  composites to optimize fracture toughness and stress resistance.

Figure 2 shows three  $\text{ZrO}_2$  crystal structures that can transform with changes in density, depending on the temperature range. Maintaining  $\text{ZrO}_2$  in a tetragonal phase at room temperature enhances its mechanical and aesthetic properties. Under stress, the material undergoes martensitic transformation, which helps absorb stress and improve crack resistance through transformation toughening. However, the  $K_{IC}$  of 3Y-TZP is moderate ( $4\text{--}5 \text{ MPam}^{1/2}$ ), and it is susceptible to low-temperature degradation (LTD) [17, 18].



Fig. 1. Applications of 3Y-TZP in dental restorations (a) Full crown; (b) Inlay and Onlay; (c) Abutment and Implant

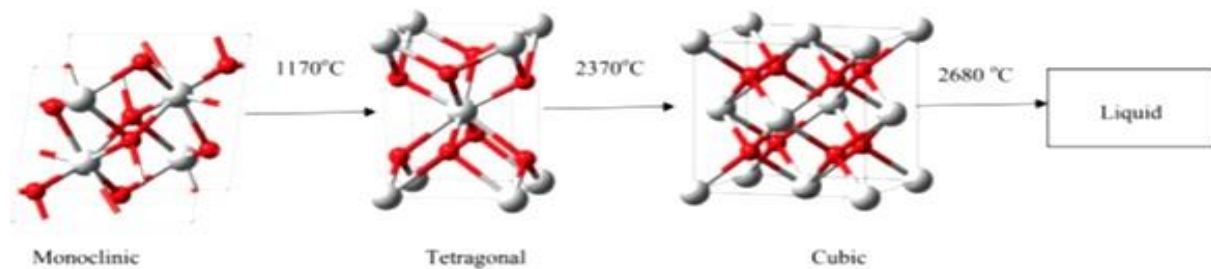


Fig. 2. Crystal structure of three monoclinic, tetragonal, and cubic phases and their transformation temperature

Rietveld analysis is a powerful and widely used technique in the field of crystallography. It is a method for refining crystal structures from X-ray or neutron diffraction data. Rietveld analysis allows researchers to determine the precise atomic arrangement within a crystal lattice by comparing experimental diffraction patterns with theoretical patterns calculated from a structural model [19].

The issue of failure in brittle materials has always been pertinent in engineering practice. One prevalent form of mechanical failure is known as brittle fracture [20, 21]. The weakening and abrupt collapse of the structure can occur as a result of mechanical failure, occlusal loading, and imperfections in dental materials. [22]. Restorative brittle materials used in dental treatments can contain small-sized defects such as micro-cracks and micro-porosities, which are randomly dispersed [23, 24]. The presence of these defects can greatly impact the way macro-cracks propagate. The interaction between macro and micro-cracks has been a focus of several experimental investigations in the literature

[25, 26]. Various studies indicate that the presence of micro-cracks can result in a phenomenon known as “micro-crack toughening” [27]. Micro-cracks can serve as inhibitors of crack propagation and play a role in expanding the fracture zone [28, 29]. Moreover, the stress shielding effects resulting from the interaction between micro and macro cracks can depend on factors such as their distribution, distances, sizes, and inclination angles. This interaction can cause a decrease in the stress intensity factor (SIF) [30].

Numerous analytical studies have been conducted to explore various scenarios, such as the presence of a micro-crack positioned ahead of the primary crack [31], the primary crack's interaction and various arrays of micro-cracks [32], and the distribution of micro-cracks surrounding the main crack [33]. These studies entail examining the stress fields near the crack tip, which are defined by parameters such as  $K_{IS}$  for models featuring a macro-crack and  $K_{ISIF}$  for diverse configurations of micro-cracks. The interaction between the macro-crack and micro-

cracks is quantified utilizing the  $K_I/K_0$ , which helps determine whether micro-cracks promote or hinder the propagation of the macro-crack and offers insights into the complex structural response in the presence of defects. Moreover, when a plate contains both macro-cracks and micro-cracks, the presence of micro-cracks can influence the behavior of the primary crack [34, 35]. In certain cases, the primary crack may align with the orientation of the micro-cracks, indicating the impact of micro-cracks on the propagation characteristics of the main crack. Traditional continuum mechanics-based methods have been utilized to tackle the intricate issues related to crack interactions. However, these methods often involve simplifications and assumptions to facilitate problem-solving. For instance, mathematical computations frequently concentrate on the relationship between the primary crack and micro-cracks, disregarding the possible mutual interactions among the micro-cracks themselves [27, 34].

Some studies focusing on the interaction between cracks and holes such as porosities have revealed the occurrence of momentary crack arrest phenomena [36].

These investigations have demonstrated that in dental restoration materials with micro-porosity defects, crack propagation can be momentarily interrupted before resuming with increased speed. The location and size of the defects, as well as the loading rates, determine whether the crack can be stopped or continue through the porosities. When a micro-porosity is positioned off-center from the main crack, the crack deflects towards it before returning to its original path [37]. This behavior suggests that the presence of micro-porosity can affect the path of crack propagation. Milios and Spathis [38] examined how the imbalance of the stress field near a micro-hole can lead to the curvature of a propagating crack, resulting in crack arrest. Additionally, the variation in the  $K_I/K_0$  was explored in [39], highlighting that the SIF values increase during the crack arrest phase. This indicates that changes in SIF values influence the halting of crack propagation. An integral equation method was suggested to investigate the configurations of voids, including holes, porosities, and cracks [40]. These studies showed that the presence of porosities may result in toughening effects within the material structure. This underscores the importance of considering the sizes and distribution of porosities, as they significantly impact stress mitigation or amplification. Therefore, meticulous attention to micro-porosity sizes and their spatial distribution is essential for understanding the overall performance and durability of dental ceramics. The topic of structural defects has been

a focal point in the literature. Although experimental fracture analyses are beneficial, they can be expensive and demand meticulous control to guarantee precision.

Numerical tools provide a valuable alternative for investigating fracture behavior and mechanisms, offering a way to overcome the limitations and expenses associated with experimental testing. The finite element method (FEM) has been utilized for predicting brittle fracture, simulating crack propagation, and modeling crack branching. FEM is based on classical continuum mechanics equations expressed as partial differential equations. To simulate crack propagation accurately, Tracking the crack surface, determining crack growth direction, and assessing the SIF are crucial for understanding crack propagation and material failure [41]. In addition, FEM is mesh-dependent, necessitating model re-meshing after each propagation step to address crack growth challenges. To overcome these issues, the extended finite element method (XFEM) was developed to handle problems involving discontinuities like cracks [42]. In XFEM, cracks are modeled independently of the mesh, leading to more precise simulations. XFEM has proven effective in various scenarios involving interactions between macro- and micro-cracks [43]. However, determining the shape and position of the crack tip remains a critical aspect that requires careful consideration.

As an alternative to classical continuum mechanics, Silling [44] introduced a novel numerical approach for fracture analysis known as peridynamics (PD). PD models employ integro-differential equations rather than partial differential equations, enabling the examination of intricate crack interaction scenarios without the necessity of tracking crack tip positions or behavior. The original “bond-based” PD formulation has proven to be effective in addressing brittle fracture issues by accurately forecasting crack initiation, propagation [45-48], and the interaction between primary cracks and micro-cracks [49, 50]. The commonly used “bond-based” PD model has limitations in defining material properties, as it enforces Poisson’s ratio to 1/3 in 2D models and 1/4 in 3D models [51]. To address this constraint, Silling *et al.* [52] introduced the ordinary state-based PD method. This enhanced model allows for varying Poisson’s ratios in 2D and 3D models, offering more realistic outcomes compared to the restricted 1/3 and 1/4 ratios [26].

This study presents a comprehensive examination of PD in dental restorations for the first time. It investigates the effects of nanoporosity and micro-cracks on the propagation of macro-cracks in 3Y-TZP ceramics

with low  $\text{Al}_2\text{O}_3$  content, an area that has not been extensively explored by researchers. The research integrates experimental data with results from PD and FEM theories to assess how these factors affect crack propagation behavior in dental ceramics. Various 3Y-TZP dental ceramics were synthesized with  $\text{Al}_2\text{O}_3$  doping levels of 0, 0.02, 0.05, 0.1, 0.2, 0.25, 0.3, and 0.5 wt% through high-energy ball milling of  $\text{ZrO}_2$  powders. The mechanical properties and LTD of the dental plate samples were evaluated. A brief introduction to ordinary state-based PD is provided, and PD simulations are employed to explore the influence of nanoporosities and micro-cracks on macro-crack propagation. The critical loads for five different scenarios involving micro-crack presence are compared with analytical solutions.

## 2. PD Formulation in Dental Plate Ceramics

The state-based peridynamic method, introduced by Silling and Askari [53], presents a redefined set of equations from continuum mechanics that is particularly adept at handling issues related to discontinuities. PD utilizes integro-differential equations, a departure from the partial differential equations used in classical continuum mechanics. This expanded framework allows for the resolution of fracture mechanics challenges, including the onset and advancement of cracks. The equation of motion in PD can be represented as an integrodifferential equation, as in equation 1, offering a unique approach to modeling material behavior [54]:

$$\rho \ddot{u}(x, t) = \int_H \left( t(u' - u, x' - x, t) - t(u - u', x - x', t) \right) dV' + b(x, t) \quad (1)$$

where  $H$  is a neighborhood of  $x$ ,  $u$  is the displacement vector field,  $b$  is a prescribed body force density field,  $\rho$  is mass density in the reference configuration, and  $t$  is a pairwise force

function whose value is the force vector that the particle  $x'$  exerts on the particle  $x$ .

The above equation can be discretized as shown in equation 2 [26]:

$$\rho_{(i)} \ddot{u}_{(i)} = \sum_{j=1}^N \left( t_{(i)(j)} \left( u_j - u_i, x_{(j)} - x_{(i)}, t \right) - t_{(j)(i)} \left( u_{(i)} - u_{(j)}, x_{(i)} - x_{(j)}, t \right) \right) V_{(j)} + b_{(i)} \quad (2)$$

The acceleration of material point  $i$  at time  $t$ , denoted as  $\ddot{u}_{(i)}$ , can be obtained through discretization in dental ceramics. Material point  $i$  interacts with other points  $j$  within its horizon  $H_{x(i)}$ , with a total of  $N$  family members for point  $i$ . This interaction is shown in Figure 3.

The material point's position is marked by  $x$  in a volume  $V$ . The displacement field, body load, and mass density are denoted as  $u$ ,  $b$ , and  $\rho$ . In dental ceramic plate simulations, the horizontal and vertical displacements are represented by  $u$  and  $v$ .

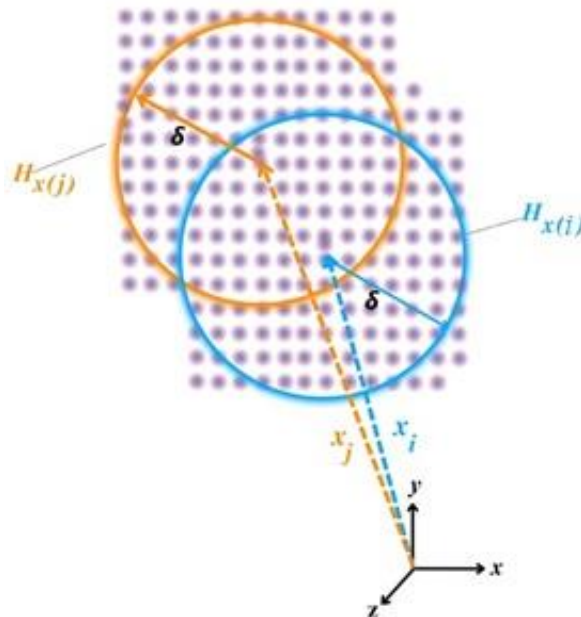


Fig. 3. Peridynamic material points and their interaction within the dental plate

In a 2-D context, the interaction between material points  $i$  and  $j$  can result in unequal forces in the ordinary state-based PD method [55].

The PD force densities for points  $i$  and  $j$  are outlined in equations 3 and 4 [26]:

$$t_{(i)(j)} = 2\delta \left( d \frac{\Lambda_{(i)(j)}}{|x_{(j)} - x_{(i)}|} a\theta_{(i)} + 2\delta bs_{(i)(j)} \right) \times \frac{y_{(j)} - y_{(i)}}{|y_{(j)} - y_{(i)}|} \quad (3)$$

$$t_{(j)(i)} = -2\delta \left( d \frac{\Lambda_{(i)(j)}}{|x_{(j)} - x_{(i)}|} a\theta_{(j)} + 2\delta bs_{(i)(j)} \right) \times \frac{y_{(j)} - y_{(i)}}{|y_{(j)} - y_{(i)}|} \quad (4)$$

here,  $\delta$  is the horizon size, and the parameters  $a$ ,  $b$ , and  $d$  are unique to the PD method. The distance between material points is shown as  $s$ , and the dilatation, denoted as  $\theta$ , can be expressed as equation 5 [26]:

$$\theta_{(i)} = d \sum_{j=1}^N \Lambda_{(i)(j)} s_{(i)(j)} V_{(j)} \quad (5)$$

$\Lambda_{(i)(j)}$  is defined in equation 6 [26]:

$$\Lambda_{(i)(j)} = \left( \frac{y_{(j)} - y_{(i)}}{|y_{(j)} - y_{(i)}|} \right) \left( \frac{x_{(j)} - x_{(i)}}{|x_{(j)} - x_{(i)}|} \right) \quad (6)$$

In the altered state, the updated position of a material point post-deformation is denoted as  $y$ , as illustrated in Figure 4.

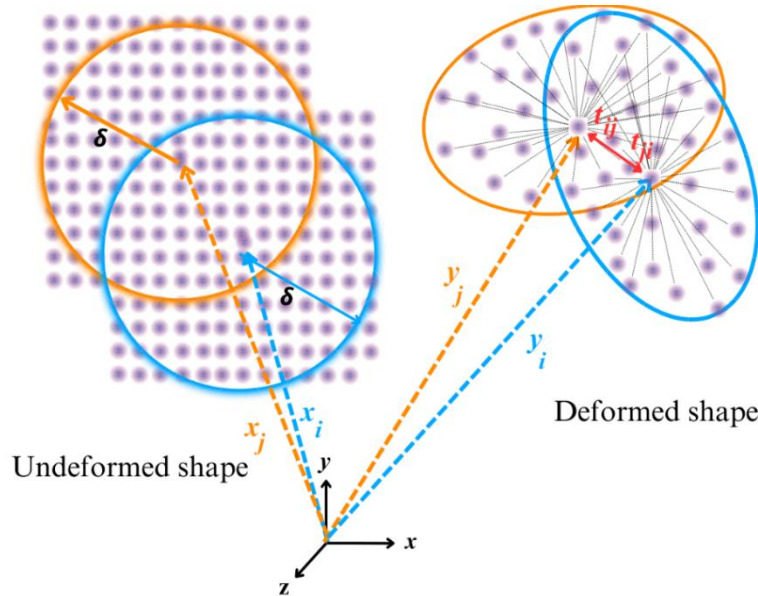


Fig. 4. Peridynamics parameters in undeformed and deformed states

The PD parameters  $a$ ,  $b$ , and  $d$  can be determined by equating the strain energy density of a material point inside a body under isotropic expansion and simple shear loading conditions, as calculated using classical continuum mechanics and PD [55]. This allows for a relationship to be established between these PD parameters and the material constants of classical continuum mechanics. This study uses a 2-D model for 3Y-TZP/Al<sub>2</sub>O<sub>3</sub> dental materials in plane stress conditions. The plate is discretized with a single layer in the thickness direction. This allows the expression of the PD parameters as the bulk modulus ( $\kappa$ ), thickness ( $h$ ), shear modulus ( $\mu$ ), and horizon size ( $\delta$ ) for the 2-D problem presented in equation 7 [55]:

$$a = \frac{1}{2}(\kappa - 2\mu), \quad b = \frac{6\mu}{\pi h \delta^4}, \quad d = \frac{6\mu}{\pi h \delta^3} \quad (7)$$

This modeling method is important as dental restorative materials such as crowns, inlays, onlays, abutments, and veneers on implants often encounter surface tension during chewing. Therefore, the tension between dental material points  $i$  and  $j$  can be described in equation 8 [26]:

$$s_{(i)(j)} = 1 - \frac{(|y_j - y_i| - |x_j - x_i|)}{|x_j - x_i|} \quad (8)$$

Silling and Bobaru [56] introduced a failure parameter that incorporates a history-dependent scalar-valued function  $\mu$ . This function is used to represent broken interactions (bonds) between material points, which can be expressed in Equation 9 [56]:

$$\mu(x_{(j)} - x_{(i)}, t) = \begin{cases} 1 & s_{(i)(j)}(x_{(j)}, x_{(i)}, t') < s_c \quad 0 < t' \\ 0 & \text{otherwise} \end{cases} \quad (9)$$

In equation 9,  $S_c$  denotes the critical stretch, which can be defined for a 2-D scenario in equation 10 [55]:

$$s_c = \sqrt{\frac{G_c}{\left(\frac{6}{\pi}\mu + \frac{16}{9\pi^2}(\kappa - 2\mu)\right)\delta}} \quad (10)$$

In this equation, the critical energy release rate is represented by  $G_c$ . The local damage of material point  $i$ , ranging from 0 to 1, can be expressed in equation 11 [26]:

$$\varphi(x_i, t) = 1 - \frac{\sum_{j=1}^N \mu(x_{(j)} - x_{(i)}, t) V_{(j)}}{\sum_{j=1}^N V_{(j)}} \quad (11)$$

### 3. Materials and Methods

#### 3.1. Samples Preparation

Eight different compositions of dental ceramics were investigated. The  $ZrO_2$  powder used in the experiment was prepared through a solid-state reaction high-energy ball milling method.  $ZrO_2$  (Hang-Zhou Wan-Jing Co., AR Grade, purity  $\geq 99.98\%$ ,  $500 \pm 100$  nm) and  $Y_2O_3$

(Shang-Hai Yue-Kai Co., AR Grade, purity  $\geq 99.98\%$ ) were used as raw materials. The eight materials consisted of 3 mol% yttria-stabilized zirconia with varying amounts including 0, 0.02, 0.05, 0.1, 0.20, 0.25, 0.3, and 0.5 wt% of  $\alpha-Al_2O_3$  (Feng-Fan Chemical Co., Ltd., AR Grade, purity  $\geq 99.98\%$ ) added to form 3Y-TZP. The precise quantities of each component were calculated based on stoichiometry. The powders were subjected to high-energy ball milling at 3000 r/min for 2 h, followed by 2800 r/min for 2 h, and then dried completely to obtain 3Y-TZP powder. To prepare the 3Y-TZP + x wt%  $Al_2O_3$  powder, the dried powder was additionally dried at 75 °C for 24 h to eliminate any residual ethanol. The resulting powders were uniaxially pressed under 30 MPa and then cold isostatically pressed to form pellets under 200 MPa. The green pellets were sintered in a pressure-less air environment at 1500 °C for 2 h.

#### 3.2. Relative Density

Table 1 values of  $Al_2O_3$  additive of 3Y-TZP + x wt%  $Al_2O_3$  samples and their theoretical density values can be observed. The dental samples' density was determined through the Archimedes technique as outlined in ASTM C373 [57]. The theoretical density of the samples was calculated using the values of 6.1 g/cm<sup>3</sup> for 3Y-TZP [58] and 3.99 g/cm<sup>3</sup> for  $\alpha-Al_2O_3$  [58].

**Table 1.** Values of  $Al_2O_3$  additive in 3Y-TZP+ x wt%  $Al_2O_3$  dental samples and their theoretical density

Sample	3Y-TZP (wt.%)	$Al_2O_3$ (wt.%)	Theoretical density
x= 0	100.00	0.00	6.100000
x= 0.02	99.98	0.02	6.099800
x= 0.05	99.95	0.05	6.099173
x= 0.1	99.90	0.10	6.098346
x= 0.2	99.80	0.20	6.096692
x= 0.25	99.75	0.25	6.095865
x= 0.3	99.70	0.30	6.095038
x= 0.5	99.50	0.50	6.091730

#### 3.3. Microstructural

The microstructure of polished surfaces treated at 1300 °C for 40 min in air and Pt-coated surfaces was analyzed using scanning electron microscopy (FESEM, Zeiss, Sigma 500). Grain size analysis was performed on SEM micrographs utilizing the linear intercept method, with a minimum of 1200 grains counted. The reported average results ( $\pm$  SD) were provided without any modifications [59]. The bulk density of the sintered samples was determined by applying Archimedes' principle, with measurements carried out in deionized water.

#### 3.4. Aging Kinetics

The LTD of various components was investigated by subjecting polished samples to water vapor at temperatures of 140 °C for different durations (0, 20, 30, 50, 80, 100, 200, 300, 500, and 800 h) using an autoclave (FCF-50 L, Zhengzhou Yuhua Instrument Manufacturing Co.). The transformation content of all specimens was analyzed using X-ray diffraction (XRD) with Cu-K $\alpha$  radiation (50 kV, 45 mA) in the  $2\theta$  range of 29° to 32°, with a step size of 0.03 1/s. The volume fractions of m- $ZrO_2$  and t- $ZrO_2$  were determined from the diffraction patterns,

following the methodology proposed by Toraya et al. [60] and Garvie and Nicholson [61] in equations 12 and 13:

$$V_m = \frac{1.311X_m}{1 + 0.311X_m} \quad (12)$$

$$X_m = \frac{I_m(111) + I_m(111)}{I_m(111) + I_m(111) + I_t(101)} \quad (13)$$

where  $V_m$  represents the volume monoclinic phase,  $X_m$  indicates the proportion of the monoclinic phase relative to the total composition,  $I_m$  and  $I_t$  corresponds to the intensities of the diffraction peaks associated with m-ZrO<sub>2</sub> and t-ZrO<sub>2</sub> components, respectively.

Following the aging process, the aging time was used as a variable to illustrate the change curve of the monoclinic content. It is important to highlight that all samples exhibited XRD patterns between 20-80° (2θ) before aging, ensuring experiment precision. Additionally, to compare the aging kinetics across various doped ceramic samples, the transformation curves were fitted using the Mehl-Avrami-Johnson (MAJ) equation [62], as expressed in equation 14:

$$f_m = \frac{V_m}{V_{ms}} = 1 - \exp(-(bt)^n) \quad (14)$$

where  $f_m$  represents volume monoclinic fraction,  $V_m$  is monoclinic phase content,  $V_{ms}$  is the saturation of monoclinic phase,  $t$  is time,  $b$  ( $h^{-1}$ ) is the kinetic parameter of phase transition, and  $n$  is the parameter associated with phase change structure factor [63].

### 3.5. Methods of Determining Mechanical Properties

The Nanoindentation XP device from the Nanoinstruments Innovation Center (MTS systems, TN, USA) was utilized for nanoindentation experiments. Equipped with a Berkovich diamond indenter featuring a 50 nm tip radius, the tests presented data in the form of graphs showcasing the applied force ( $P$ ) and penetration depth ( $h$ ) throughout the loading and unloading phases [64]. In these experiments, a maximum load of 780 mN was applied, resulting in a maximum penetration depth of around 1180 nm in the material. All tests were carried out at room temperature, with the system being calibrated using a fused silica standard prior to the experiments. Analyzing the force-depth curves derived from the nanoindentation tests, the hardness and Young's modulus ( $E$ ) values of

the samples were determined relative to penetration depth, with five measurements conducted for accuracy. For the analysis of the graphs, the nanoindentation test of the force component ( $P$ ) varying with the indented depth component ( $h$ ) during loading and unloading is plotted, and equations 15 to 17 are used [64]:

$$H_v = \frac{P_{max}}{A_c} \quad (15)$$

$$E = \frac{\sqrt{\pi}}{2\sqrt{A}} \times \frac{dp}{dh} \quad (16)$$

$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu_i^2)}{E_i} \quad (17)$$

These equations feature  $P_{max}$ ,  $A_c$ , and  $H_v$ , which correspond to the maximum loading, contact area, and Vickers hardness, respectively. In addition,  $dp/dh$  represents the material stiffness, while  $E_r$  signifies the calculated modulus.  $A$  represents the area of all formed micro-cracks around the indented zone,  $\nu$  denotes Poisson's ratio, and the index  $i$  specifies the indenter. The Poisson's ratio and  $E$  of the diamond indenter (0.07 and 1140 GPa, respectively) are symbolized as  $\nu_i$  and  $E_i$ , whereas the sample's corresponding properties are denoted as  $\nu$  and  $E$  [64, 65]. Moreover, the Vickers microhardness test, ASTM E384 [66], was employed to evaluate and compare the hardness values. In this process, a Vickers diamond load equivalent to 98.06 N was exerted on each sample for 10 sec. The Vickers microhardness test took place in a controlled laboratory environment utilizing a Tukon 2100B hardness tester device, and the average hardness value for each sample was calculated based on seven measurements. In addition, the  $E$  of each sample was determined based on the variations in sonic velocity within the sample, as outlined in equation 18 of ASTM C769-98 [67]:

$$E = \rho v^2 \quad (18)$$

where  $\rho$  represents sample density and  $v$  is the speed of sound in the sample. This method involves transmitting and receiving a 5 MHz ultrasonic pulse to and from each sample eight times to determine the speed of sound through the specimen by recording pulse times and measuring sample thickness. To determine Poisson's ratio of dental samples, equation 19 is used [65]:

$$H_v = 2 \left( \frac{9E(1-2\nu)^2}{8(1+\nu)^3} \right)^{0.585} - 3 \quad (19)$$

where  $\nu$  is dimensionless, and  $H_v$  and  $E$  are expressed in GPa. During instrumented indentation testing, the Berkovich indenter is a

popular choice because it shares a depth-to-area relationship with the four-sided Vickers pyramid commonly employed in micro-hardness evaluations. The Berkovich geometry stands out due to its three-sided pyramid shape, which can be sharpened to a point more effortlessly, retaining its self-similar geometry even at smaller scales.

In the analysis of the samples,  $K_{IC}$  values were acquired utilizing the single-edge laser-notch beam (SELNB) technique. This method involves the precision creation of an ultra-sharp V-notch on the samples using laser pulses. Subsequently, the fracture toughness of the samples is evaluated through three-point bending (TPB) tests, as detailed in [65]. To address the difficulties in machining a notch with precise depth and to prevent unintended crack formation, infrared ultra-short laser pulses were

employed to create the ultra-sharp V-notch on the samples. The notches' depths varied from 80 to 120  $\mu\text{m}$ , with a maximum tip radius of 2  $\mu\text{m}$ . Given that the tip radius of the notch can impact the  $K_{IC}$  values, ensuring a sharpness of 2  $\mu\text{m}$  was crucial to enhance result accuracy, as referenced in [58, 65, 68].

Figure 5 shows the created notch on the 3Y-TZP+ 0 wt%  $\text{Al}_2\text{O}_3$  sample. It can be observed that the notch has appropriate geometry, and the absence of unintended damage indicates its quality and accuracy. The samples underwent testing using a TPB machine with a gauge length of 10 mm and a consistent displacement rate of 0.1 mm/min at room temperature.

Figure 6 illustrates the schematic of the laser-notched sample for  $K_{IC}$  testing in the TPB test. Each sample underwent five SELNB tests, and the average values were recorded.

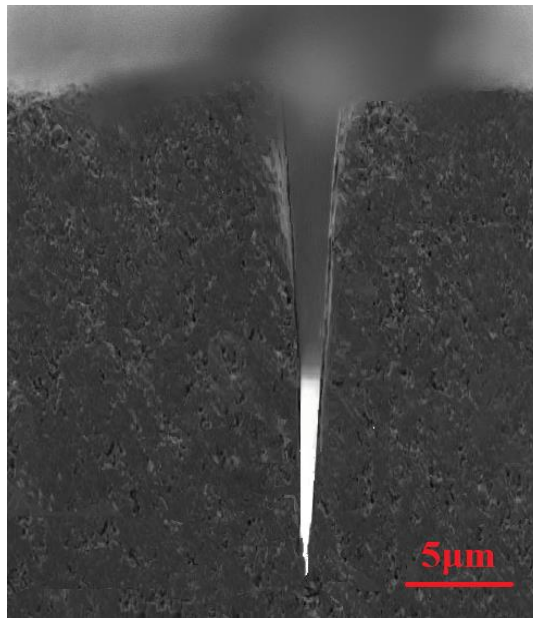


Fig. 5. Laser-produced V-notch view in 3Y-TZP+ 0 wt%  $\text{Al}_2\text{O}_3$  specimen

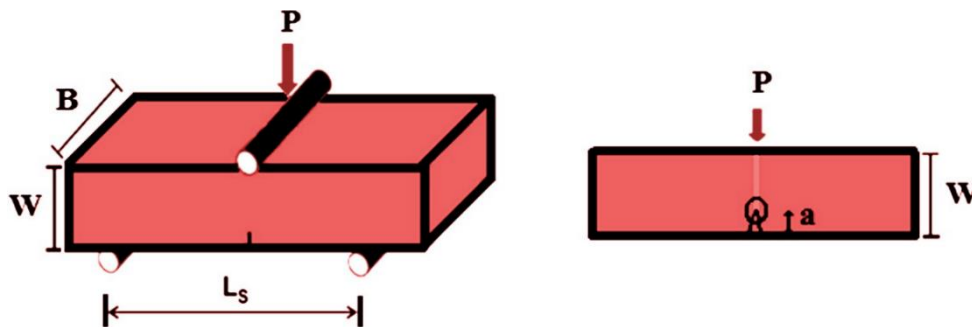


Fig. 6. Schematic of a Laser-notched plate dental sample in the SELNB test

Equations 20 and 21 were applied in this method to determine the  $K_{IC}$  values, as described in reference [69]:

$$K_{IC} = \frac{3L_s}{2BW^{\frac{3}{2}}} \cdot P_{\max}^{\frac{1}{2}} \cdot Y^* \quad (20)$$

where  $P_{\max}$  is the applied load at fracture,  $B$  represents the width of the specimen bar,  $W$  denotes the thickness of the specimen bar,  $a$  is the notched depth ( $\frac{a}{W}$ ).

$$Y^* = \frac{(1.989 - 0.356\beta) - (1.217 + 0.315\beta)\alpha + (3.212 + 0.705\beta)\alpha^2 - (3.222 + 0.02\beta)\alpha^3 + (1.226 - 0.015\beta)\alpha^4}{(1-2\alpha)(1-\alpha)^{\frac{3}{2}}} \quad (21)$$

$Y^*$  is the stress intensity shape factor, and  $\beta = \frac{W}{L_s}$ .

The flexural strength (*MOR*) of the samples is determined using the TPB method ASTM C1161 [70] and calculated based on equation 22. Ten measurements are taken for each sample, and the average value is obtained.

$$MOR = \frac{3Fl}{2bd^2} \quad (22)$$

where  $F$  represents the fracture force (N),  $l$  is the support span (m),  $b$  is the width of the sample (m), and  $d$  is the thickness of the sample (m).

## 4. Results and Discussion

### 4.1. Microstructure and Aging Resistance

The XRD pattern in Figure 7 displays the analysis of 3Y-TZP +  $x$  wt%  $Al_2O_3$  ceramics after aging for 0 h and sintering in air at 1500 °C for 2 h. All eight 3Y-TZP/ $Al_2O_3$  samples exhibited similar XRD patterns, showing distinct peak

characteristics. The absence of monoclinic peaks ( $-111$  m and  $111$  m at  $2\theta$  values of  $28.3^\circ$  to  $31.4^\circ$ ) was observed. In the  $76-79^\circ$  ( $2\theta$ ) range, the cubic phase peak (400) c was split into two peaks of the tetragonal phase, (004) t and (400) t. The presence of the characteristic (400) c peak and the cubic phase between the tetragonal (200) t and (002) t peaks was notably absent. Jade analyses indicated that the 3Y-TZP +  $x$  wt%  $Al_2O_3$  ceramics ( $x = 0, 0.02, 0.25, 0.1, 0.2, 0.25, 0.3$ , and  $0.5$ ) comprised approximately 95% tetragonal phase and 5% monoclinic phase. This composition suggested that  $Al_2O_3$  and  $Y_2O_3$  had partially dissolved into the zirconia matrix, leading to  $Y_3^+$  and  $Al_3^+$  segregation at the grain boundaries, which is in line with established theories [71]. It is observed that increasing the proportion of the  $Al_2O_3$  phase leads to an increase in the amount of monoclinic phase within the existing  $ZrO_2$  structure. The highest quantity of monoclinic phase is attained in the 3Y-TZP sample without  $Al_2O_3$ .

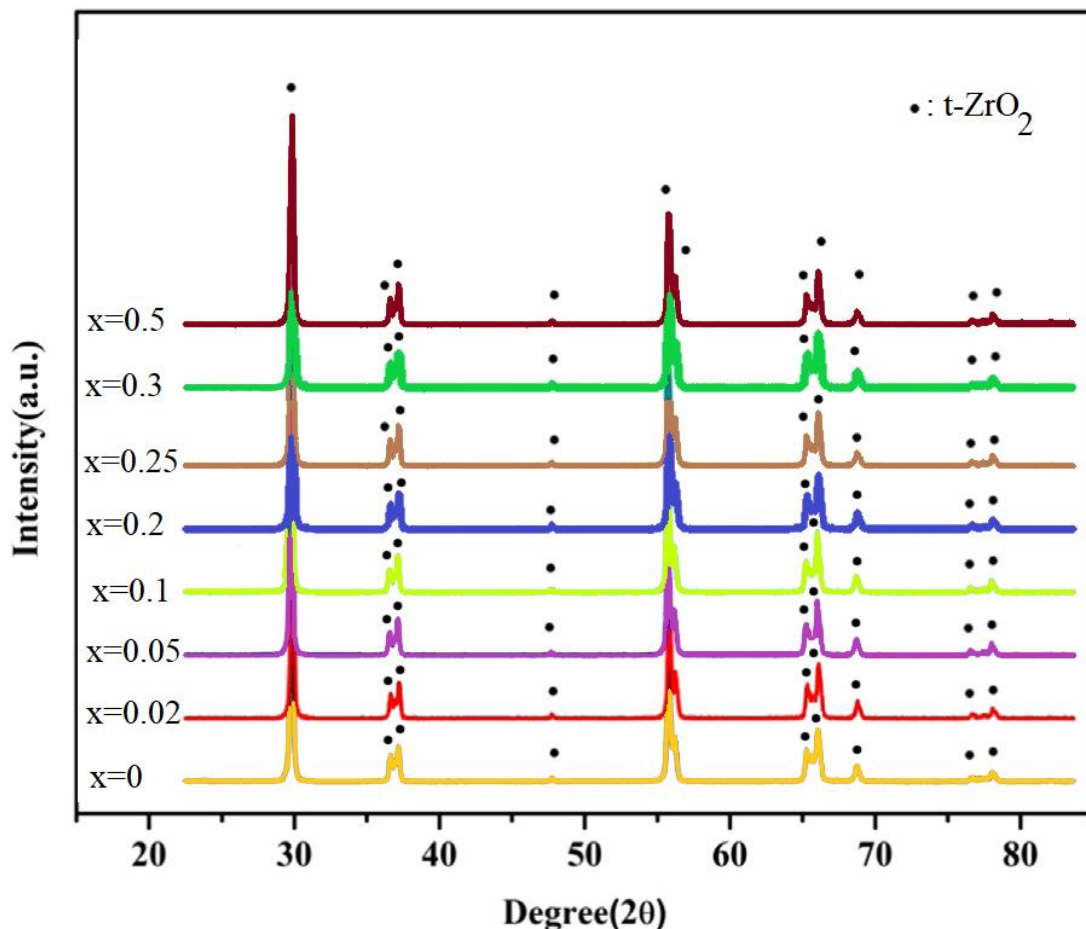


Fig. 7. XRD pattern of the 3Y-TZP +  $x$  wt%  $Al_2O_3$  dental ceramics at 0 h of aging

Figure 8-a illustrates the surface monoclinic phase content of the 3Y-TZP + x wt%  $\text{Al}_2\text{O}_3$  ceramics after sintering via a pressure-less method for 4 h at 1500 °C over a duration ranging from 0 to 800 h. In the case of 3Y-TZP ceramics doped with  $\text{Al}_2\text{O}_3$ , the content of m-ZrO<sub>2</sub> increases following the typical "S" curve pattern when plotted against the logarithm of aging time. This growth process can be divided into three main stages. Initially, there is a slow increase in monoclinic phase content with aging time, followed by a rapid escalation in the second stage, and ultimately reaching a saturation point in the third stage. Low-temperature aging leads to degradation, and the "S" shaped degradation observed in the ceramic material is a result of the isothermal transformation from the tetragonal to monoclinic phase. The hydrothermal stability improves with increasing  $\text{Al}_2\text{O}_3$  content due to the enhancement of  $E$ , which hinders the growth of tetragonal phase ZrO<sub>2</sub> grains, contributing to stability. In addition,  $\text{Al}_2\text{O}_3$  is more prone to hydrolysis than Y<sub>2</sub>O<sub>3</sub>, allowing for the formation of a protective matrix on the material surface.

Figure 8-b illustrates the behavior of 3Y-TZP + x wt%  $\text{Al}_2\text{O}_3$  ceramics, and by analyzing the corresponding plots of  $\ln(\ln(1/(1-f)))$  vs.  $\ln(t)$  based on the data from (a) along with linear fittings, the parameter  $n$  in the MAJ Eq. 14 can be determined. Comparing experimental data with simulations, the exponent  $n$  for 3Y-TZP + x wt%  $\text{Al}_2\text{O}_3$  ceramics falls within the range of 0.7 to 1. Upon fitting the data from Table 2, the actual  $b$  ( $\text{h}^{-1}$ ) value for the 3Y-TZP + x wt%  $\text{Al}_2\text{O}_3$  ceramic was found to be 0.020  $\text{h}^{-1}$ . This minimal  $b$  ( $\text{h}^{-1}$ ) value indicates that  $\text{Al}_2\text{O}_3$  doping can significantly reduce aging kinetics. Furthermore, the value of  $n$  increases from 0.7 to 1 with higher  $\text{Al}_2\text{O}_3$  doping levels in dental ceramics. The value of  $n$  is associated with nucleation and growth during aging, where a smaller  $n$  value suggests dominant nucleation. The small experimental  $n$  value suggests the appearance of new nucleation sites during aging, hindering nucleus growth due to interactions with the new nucleation sites. Table 2 presents the values of MAJ Eq. parameters obtained through linear regression of monoclinic phase content against time, along with other relevant data for 3Y-TZP + x wt%  $\text{Al}_2\text{O}_3$  samples.

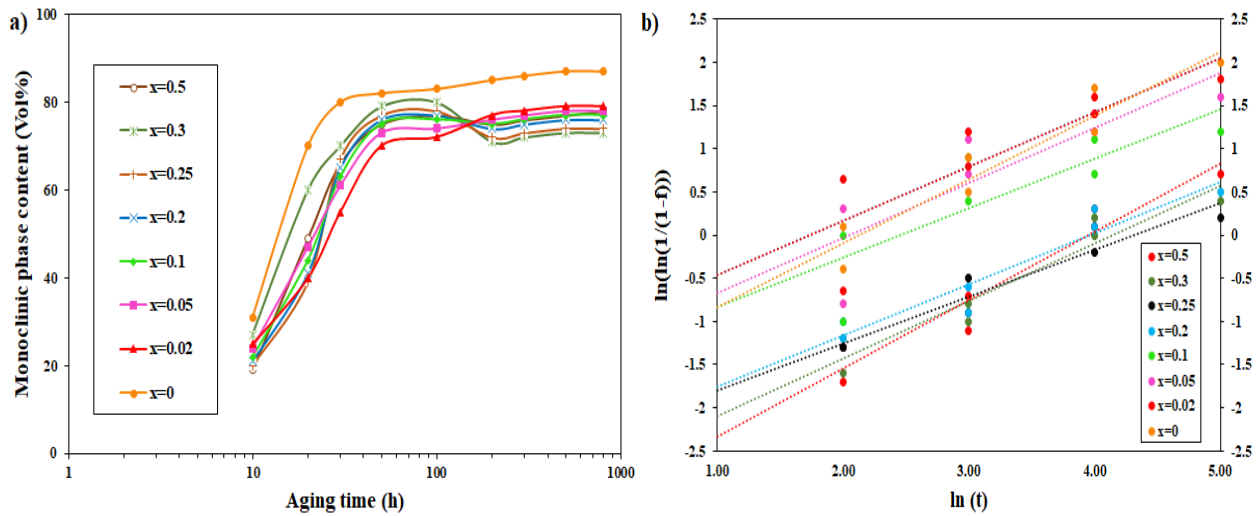


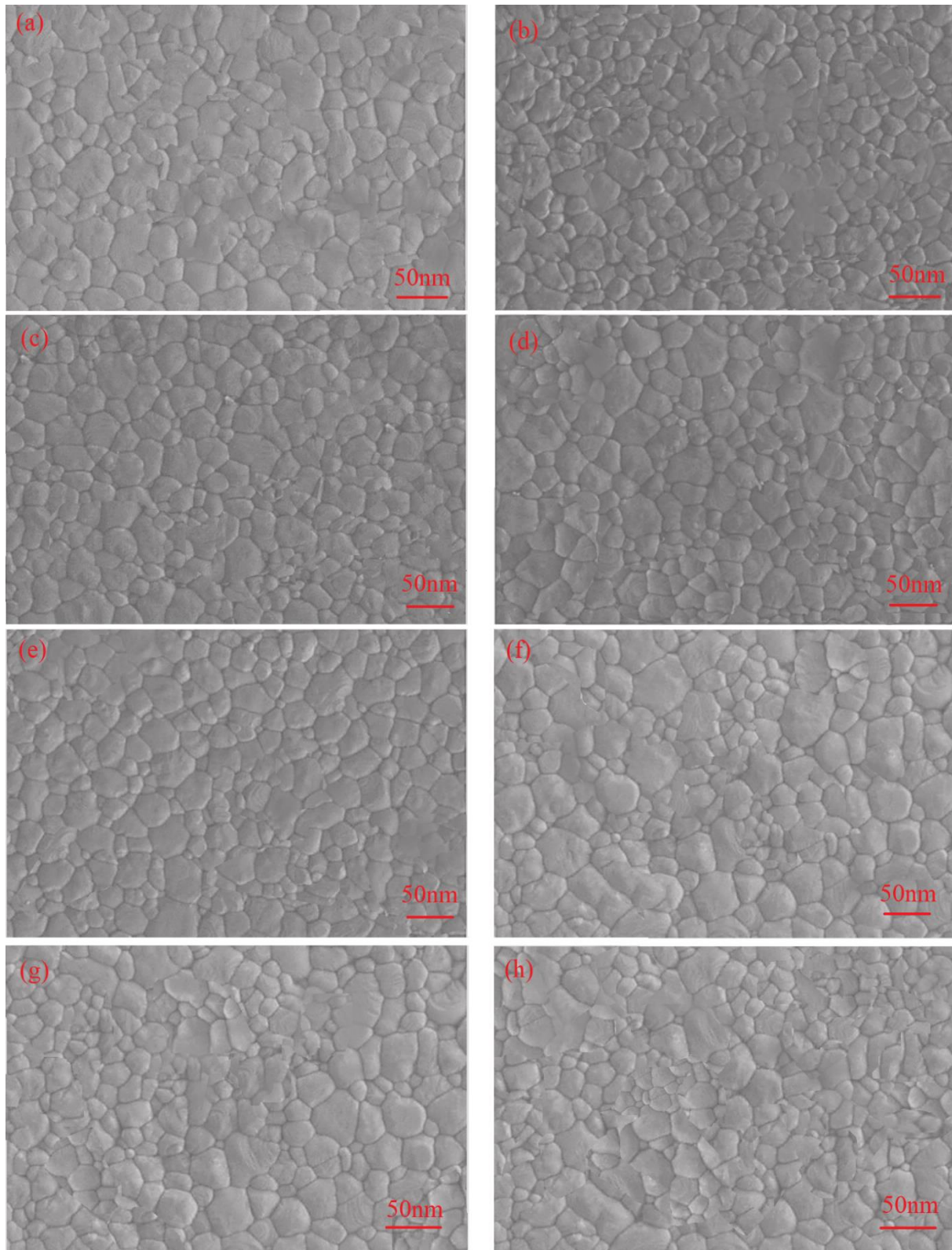
Fig. 8. (a) The percentage of surface monoclinic phase in the 3Y-TZP + x wt%  $\text{Al}_2\text{O}_3$  ceramics after pressureless sintering for 3 hours at 1450 °C following 800 h of accelerated aging at 134 °C; (b) Representation of  $\ln(\ln(1/(1-f)))$  versus  $\ln(t)$  for the 3Y-TZP + x wt%  $\text{Al}_2\text{O}_3$  ceramics, based on the results in (a), including the linear regressions employed to calculate the parameter  $n$  in equation 14 of the MAJ model

Table 2. Parameters of the MAJ equation obtained through linear regression analysis of monoclinic phase content against time in 3Y-TZP + x wt%  $\text{Al}_2\text{O}_3$

Sample	c (Å)	$V_{ms}$ (%)	$n$	$b$ ( $\text{h}^{-1}$ )
x= 0	5.2508	84.3	0.704 (0.121)	0.060 (0.014)
x= 0.02	5.2539	79.8	0.892 (0.124)	0.039 (0.007)
x= 0.05	5.2547	78.2	0.961 (0.122)	0.027 (0.005)
x= 0.1	5.2555	74.5	1.001 (0.119)	0.020 (0.003)
x= 0.2	5.2564	73.8	1.017 (0.129)	0.024 (0.009)
x= 0.25	5.2673	72.5	0.978 (0.125)	0.026 (0.004)
x= 0.3	5.2681	71.2	0.969 (0.124)	0.027 (0.005)
x= 0.5	5.2689	70.2	0.964 (0.125)	0.029 (0.003)

Figure 9 showcases typical FESEM images of the sintered 3Y-TZP + x wt%  $\text{Al}_2\text{O}_3$  ceramics, revealing a consistent microstructure across all samples. The specimens display a compact polygonal matrix with well-defined grain

boundaries. With higher  $\text{Al}_2\text{O}_3$  concentrations, the grain size shows enhanced uniformity, with the smallest and most homogeneous grain size observed at an  $\text{Al}_2\text{O}_3$  doping level of 0.25 wt%.



**Fig. 9.** FESEM micrographs of 3Y-TZP+ x  $\text{Al}_2\text{O}_3$  dental ceramics (a) x= 0, (b) x= 0.02, (c) x= 0.05, (d) x= 0.1 (e) x= 0.2, (f) x= 0.25, (g) x= 0.3, (h) x= 0.5

Table 3 summarizes the relative density, average grain diameter, and average porosity size

of the 3Y-TZP + x wt% Al<sub>2</sub>O<sub>3</sub> ceramics sintered at 1500 °C in air for 2 h.

**Table 3.** Rietveld analysis results on the phase composition, lattice parameters, relative density, average grain and average porosity diameter in 3Y-TZP+ x wt% Al<sub>2</sub>O<sub>3</sub> dental ceramic samples

Sample	Y <sub>2</sub> O <sub>3</sub> (mol%)	Relative density (%)	Average grain (nm)	Average porosity diameter (nm)
x= 0	2.75	97.4 ± 0.5	89	10
x= 0.02	2.80	98.1 ± 0.6	85	9
x= 0.05	2.80	98.3 ± 0.4	92	10
x= 0.1	2.80	98.7 ± 0.4	100	12
x= 0.2	2.80	99.1 ± 0.3	104	11
x= 0.25	2.85	99.2 ± 0.4	106	8
x= 0.3	2.80	99.3 ± 0.4	78	11
x= 0.5	2.75	99.5 ± 0.4	80	10

#### 4.2. Mechanical Properties

Figure 10 showcases the load-displacement ( $p$ - $h$ ) curves obtained from the nanoindentation test performed on the plate samples.

Figures 11 and 12 present the hardness ( $H$ ) and Young's modulus ( $E$ ) values of the 3Y-TZP+ x wt% Al<sub>2</sub>O<sub>3</sub> samples, respectively, relative to the penetration depth. Beyond a penetration depth of 120 nm, the  $E$  and  $H$  values of all samples remain relatively constant regardless of the penetration depth. As per equation 18,  $E$  is directly linked to density; hence, a decrease in density, resulting in increased porosity, also leads to a decrease in Young's modulus. Comparing the reported Young's modulus values for ZrO<sub>2</sub> ( $E \approx 250$  GPa) [72] and Al<sub>2</sub>O<sub>3</sub> ( $E \approx 380$ –500 GPa) [73–77], and noting the significant disparity between them, the critical particle size can potentially be raised to around 1 mm; whereas, for a microstructure comprising 100% 3Y-TZP phase, this critical size is approximately 0.3 mm [78]. By enhancing the proportion of the additive phase and lowering  $E$  of resultant dental ceramics, thereby alleviating the constraints imposed by the matrix and critical size parameters, the tetragonal phase content is reduced. It is also evident that the relative densities of samples have increased with the rise in the additive phase percentage. This augmentation in relative density may be attributed to the disparate thermal expansion coefficients of the two materials, resulting in the appearance of porosity in the specimens' microstructures. Furthermore, elevating the sintering temperature enhances the relative density of the samples, potentially due to the improved sinterability of the specimens.

All ceramic samples demonstrated high density and hardness, with the fracture toughness of 3Y-TZP + x wt% Al<sub>2</sub>O<sub>3</sub> exceeding 10.07 MPam<sup>1/2</sup>. This value represents a 0.9 improvement compared to similar dental ceramics in another study [79].

In the case of all ceramics prepared using the precipitation method for powder production, some Y<sub>2</sub>O<sub>3</sub> dissolved into the ZrO<sub>2</sub> lattice during the titration process (See Table 3). Therefore, from a sintering perspective, all sample ceramics underwent a single sintering synthesis. The non-uniform distribution of Y<sub>2</sub>O<sub>3</sub> within the samples resulted in the segregation of a small quantity of Y<sub>2</sub>O<sub>3</sub>. This segregation helped disperse residual stress, strengthen the grain boundary, and impede grain growth [80]. The  $H_v$ ,  $E$ ,  $K_{Ic}$  and flexural strength values of specimens are shown in Table 4.

In the realm of zirconia-based ceramics, two established toughening mechanisms are widely recognized. These mechanisms encompass toughening via a phase transition and ferroelastic toughening associated with the non-transition tetragonal phase [81, 82]. Within the framework of the toughening mechanism, when an external force of significant magnitude is applied, the metastable tetragonal phase of the ceramic material undergoes a transition to the monoclinic phase. This phase transformation generates compressive stress at the crack tip, which helps in absorbing a portion of the fracture energy. Therefore, the crack propagation is impeded, and further growth of the crack is prevented. This mechanism plays a crucial role in enhancing the fracture toughness and durability of zirconia-based ceramics [71, 83, 84].

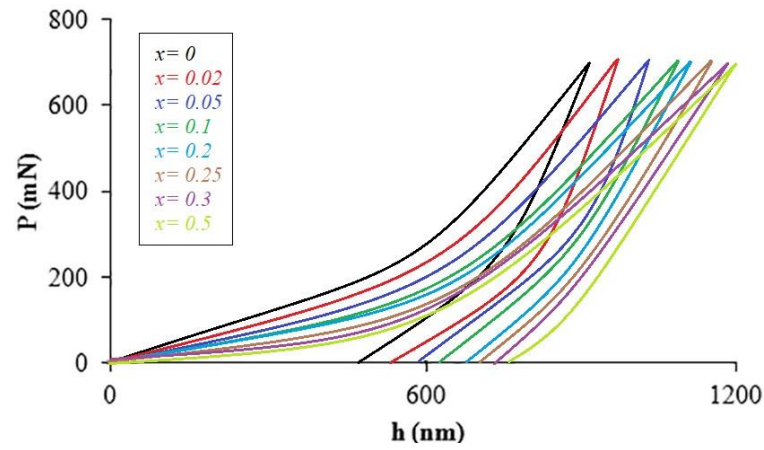


Fig. 10. Nanoindentation test results displaying the load-displacement ( $P-h$ ) curves for dental plate samples of 3Y-TZP+x  $\text{Al}_2\text{O}_3$

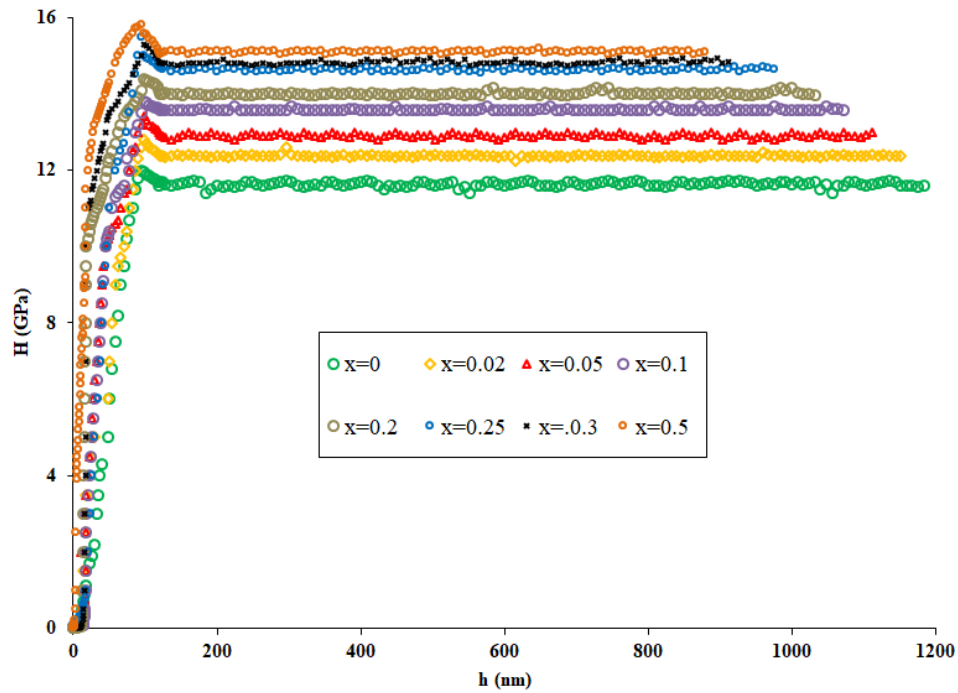


Fig. 11. Nanoindentation test results show the hardness-displacement ( $H-h$ ) curves for dental plate samples of 3Y-TZP+x  $\text{Al}_2\text{O}_3$

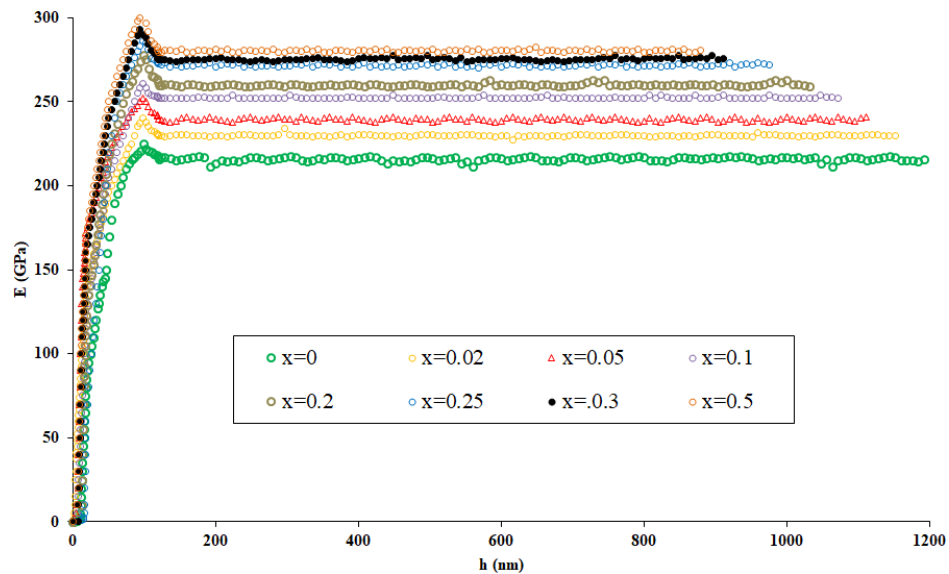


Fig. 12. Nanoindentation test results illustrate Young's modulus-displacement ( $E-h$ ) curves for dental plate samples of 3Y-TZP+x  $\text{Al}_2\text{O}_3$

**Table 4.** Hardness (GPa), Vickers hardness (GPa), Young's modulus (GPa), Poisson's ratio, Fracture toughness (MPa.m<sup>1/2</sup>), and Flexural strength (MPa) values of 3Y-TZP+ x wt% Al<sub>2</sub>O<sub>3</sub> dental plate ceramics

Sample	Nanoindentation method		Vickers Hardness indentation (GPa)	E: according to sonic velocity (GPa)	Poisson's ratio (ν)	Fracture toughness (MPa.m <sup>1/2</sup> )	Flexural strength (MPa)
	H (GPa)	E (GPa)					
x= 0	11.63± 0.33	216 ± 6.2	11.48± 0.04	211 ± 3.2	0.34	9.48 ± 0.20	312 ± 7
x= 0.02	12.38 ± 0.35	230± 6.5	11.87 ± 0.08	218 ± 6.4	0.34	9.53 ± 0.06	318 ± 6
x= 0.05	12.90 ± 0.19	240 ± 3.6	12.36 ± 0.03	232 ± 5.9	0.33	9.66 ± 0.10	321 ± 9
x= 0.1	13.59 ± 0.10	252 ± 1.9	12.78 ± 0.01	239 ± 8.3	0.32	9.72 ± 0.08	319 ± 8
x= 0.2	14.01 ± 0.21	260 ± 3.7	13.03 ± 0.02	247 ± 2.1	0.31	9.81 ± 0.07	328 ± 4
x= 0.25	14.64 ± 0.16	272 ± 3	13.33 ± 0.03	256 ± 5.7	0.30	9.91 ± 0.11	336 ± 5
x= 0.3	14.83 ± 0.20	275 ± 3.6	13.61 ± 0.02	261 ± 4.8	0.29	9.94 ± 0.09	339 ± 6
x= 0.5	15.10 ± 0.17	280 ± 3.2	14.21 ± 0.03	274 ± 9.5	0.27	9.97 ± 0.10	355 ± 7

#### 4.3. Effect of Nanoporosity on Dynamic Fracture in TPB Test

PD simulations have been carried out to examine how Young's modulus, density, Poisson's ratio, and fracture energy affect crack behavior when nanoporosities are present. Furthermore, the study delves into how the sizes of these nanoporosities impact the propagation of macro-cracks and the phenomena of crack arrest. Moreover, the analysis explores the impact of various velocity boundary conditions (BCs) on the path of the crack and its propagation speed. The appropriate time step size ( $\Delta t$ ) must be determined to ensure a stable solution. In quasi-static problems, the adaptive relaxation technique is utilized to achieve a steady-state solution, and a time step size of  $\Delta t = 1$  is used [54]. For dynamic problems involving propagating cracks, a stability criterion for  $\Delta t$  has been introduced [53]. Given that the stable time step size is contingent on the horizon size ( $\delta = 3.018 \Delta x$ ) and affected by the mass density of the 3Y-TZP/Al<sub>2</sub>O<sub>3</sub>, all dynamic computations on dental plates are executed utilizing  $\Delta t = 0.05 \mu s$ .

In Figure 13-a, a dental plate with dimensions is described as follows; length ( $L$ ) = 0.2 m, width ( $W$ ) = 0.05 m, and thickness ( $h$ ) = 0.05 m. The

dental plates are subjected to TPB loading, with supports located at a distance ( $L_s$ ) of 0.1 m from each other. At the bottom edge of the dental plate, there is a pre-existing crack with a length of  $2a = 0.004$  m. The material properties of the dental plates are described in Tables 3 and 4 for each sample. The dental plates are constrained by displacement, with  $v(x, y = W, t)$  set at  $2.5 \times 10^{-3}$  m, and have fixed supports at  $y = 0$ . The boundary conditions are implemented according to equations 23-26 [26]:

$$\begin{aligned} u_f(x_f, y_f, t + \Delta t) &= 0 \\ \text{at} \quad -2\Delta x \leq x \leq 2\Delta x \\ x_f &\in -2\Delta x \leq x \leq 2\Delta x \end{aligned} \quad (23)$$

$$\begin{aligned} v_f(x_f, y_f, t + \Delta t) &= -v(x, y, t + \Delta t), \\ W \leq y \leq W + \delta \\ y_f &\in W \leq y \leq W + \delta \end{aligned} \quad (24)$$

$$u_{f_s}(x_{f_s}, y_{f_s}, t + \Delta t) = 0 \quad (25)$$

$$v_{f_s}(x_{f_s}, y_{f_s}, t + \Delta t) = 0 \quad (26)$$

In Figure 13-b, the displacement boundary conditions are enforced at the fictitious region  $R_f$ , situated in the central part of the top edge of the plate. The PD model is discretized using  $5 \times 10^5$  material points with evenly distributed spacing between them ( $\Delta x = 6 \times 10^{-9}$  m), and a horizon size of  $\delta = 3.018 \Delta x$ . To achieve the quasi-static loading condition, a total of  $3 \times 10^3$  time steps are used with the adaptive dynamic relaxation method, and the time step size is set to  $\Delta t = 1$  s [54]. The fixed displacement and velocity

boundary conditions are rigorously established in the fictitious region  $R_{fs}$  at the bottom of the plate. The width of the  $R_{fs}$  regions precisely aligns with the horizon size of  $\delta = 3.018 \Delta x$ .

Comparisons between the PD and FEM results are meticulously examined and graphically depicted in Figures 14 and 15. Impressively, the PD findings for displacements along the central lines of the dental plates showcase remarkable consistency with the FEM outcomes.

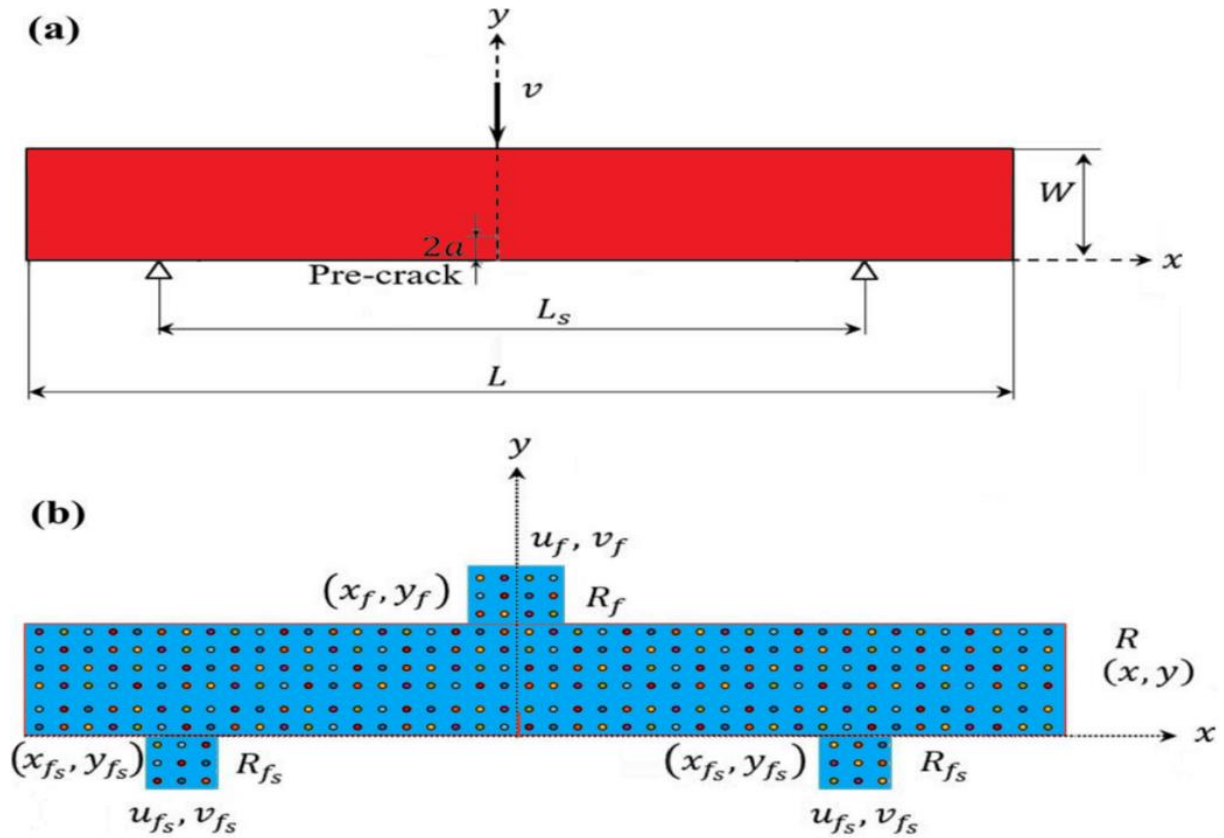
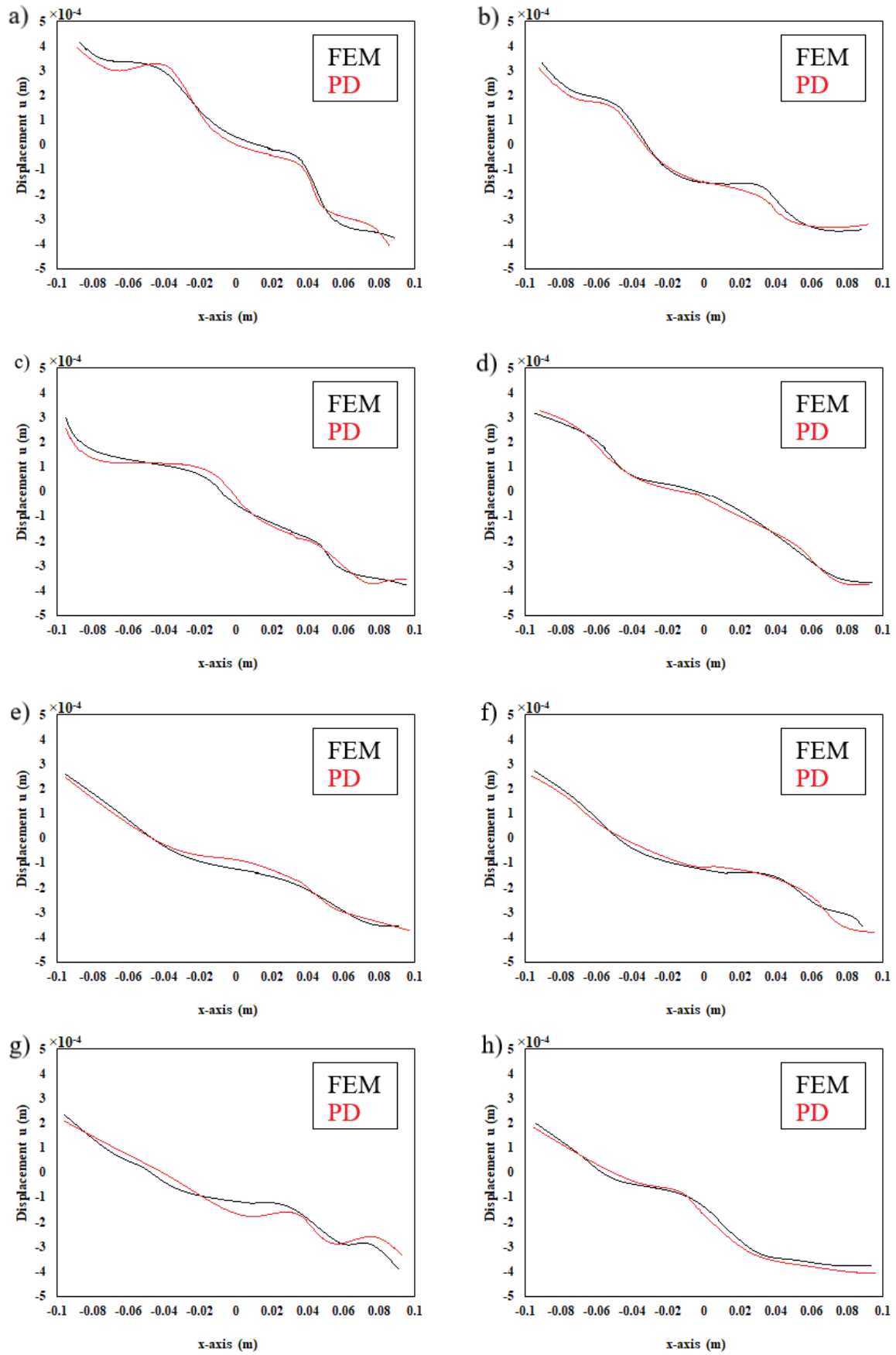
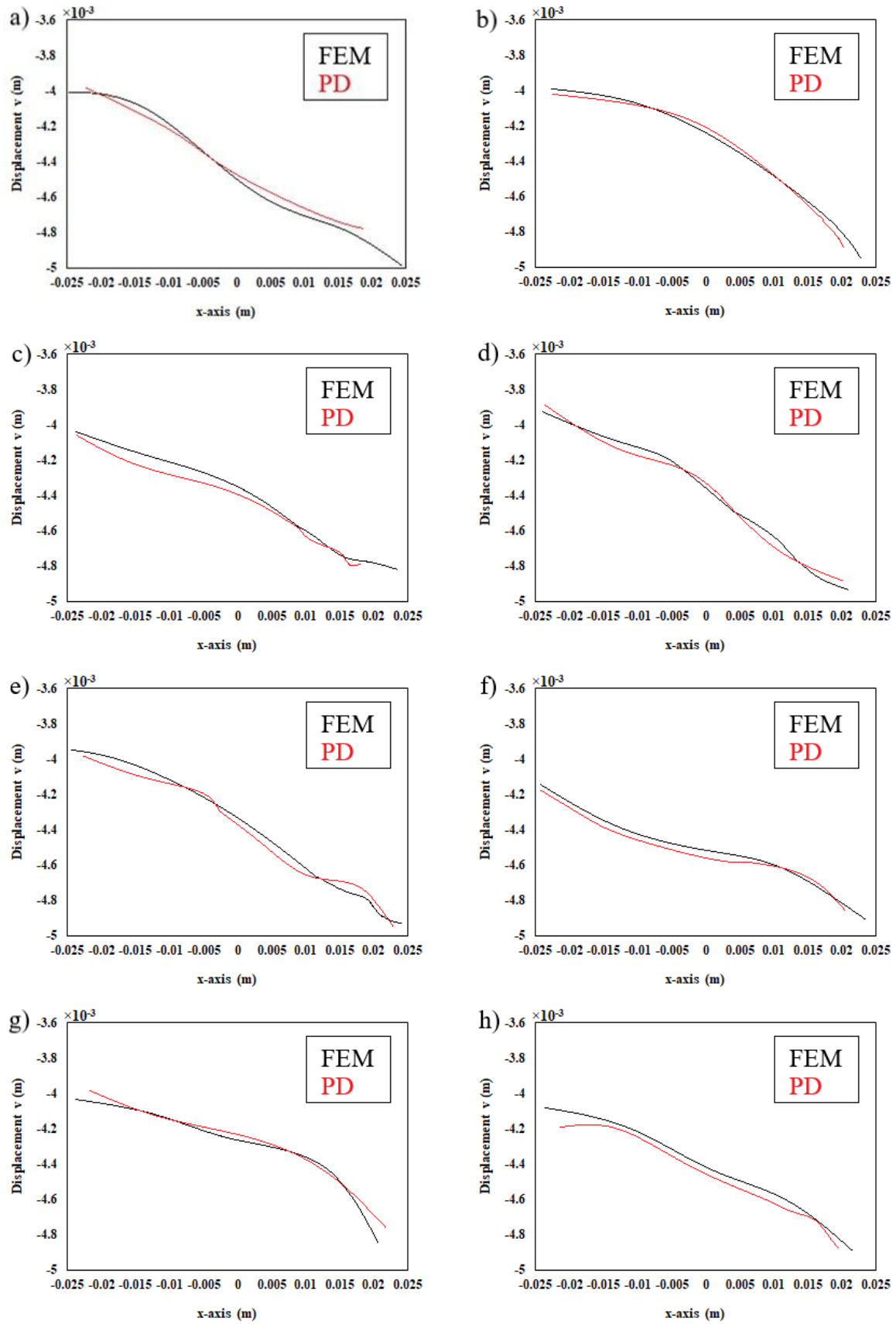


Fig. 13. a) Schematic of a pre-cracked dental plate in bending loading, b) its discretization



**Fig. 14.** Variations in displacement along the center lines in 3Y-TZP +  $x$   $\text{Al}_2\text{O}_3$  samples at  $u(x, y = 0)$  a)  $x=0$ , b)  $x=0.02$  c)  $x=0.05$ , d)  $x=0.1$ , e)  $x=0.2$ , f)  $x=0.25$ , g)  $x=0.3$  and h)  $x=0.5$  wt%



**Fig. 15.** Variations in displacement along the center lines in 3Y-TZP +  $x$   $\text{Al}_2\text{O}_3$  samples at  $v$  ( $x = 0, y$ ) a)  $x=0$  b)  $x=0.02$  c)  $x=0.05$ , d)  $x=0.1$  e)  $x=0.2$  f)  $x=0.25$  g)  $x=0.3$  and h)  $x=0.5$  wt%

According to the experimental results, the samples containing 0.1 wt%  $\text{Al}_2\text{O}_3$  exhibit the highest porosity. In Figure 16, the pre-crack in the three-point test spans a length of  $2a = 0.005$  m and is positioned at the lower edge of the plate. Following the identification of maximum porosities in the samples, an in-depth investigation is initiated. Noteworthy is the placement of the nanoporosity at a specific distance of  $dx = 0.005 + R$  m and  $dy = 0.03$  m from the origin of the main coordinate system, which is anchored at the base of the crack. Velocity boundary conditions of (a)  $\dot{v} = 1$  m/s and (b)  $\dot{v} = 4$  m/s are applied at the fictitious region located on the top edge of the plate, as visually

represented in Figure 13-b, according to equations 27 and 28 [26].

$$\begin{aligned} u_f(x_f, y_f, t + \Delta t) &= 0 \\ \text{at } -2\Delta x \leq x \leq 2\Delta x \\ x_f &\in -2\Delta x \leq x \leq 2\Delta x \end{aligned} \quad (27)$$

$$\begin{aligned} v_f(x_f, y_f, t + \Delta t) &= -\dot{v}(x, y, t + \Delta t) \times \Delta t \\ W \leq y \leq W + \delta \\ y_f &\in W \leq y \leq W + \delta \end{aligned} \quad (28)$$

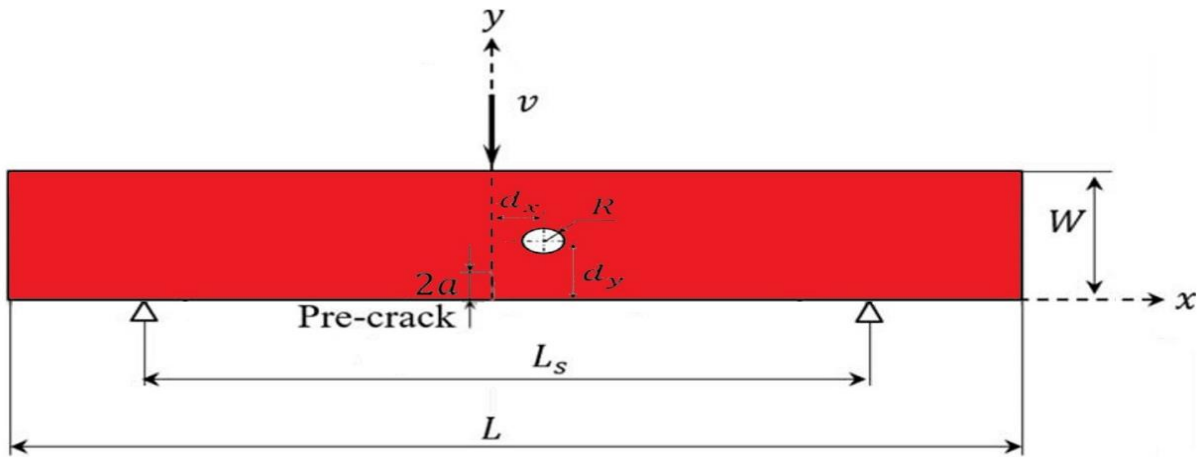


Fig. 16. Schematic of a pre-cracked dental plate with a nanoporosity in bending

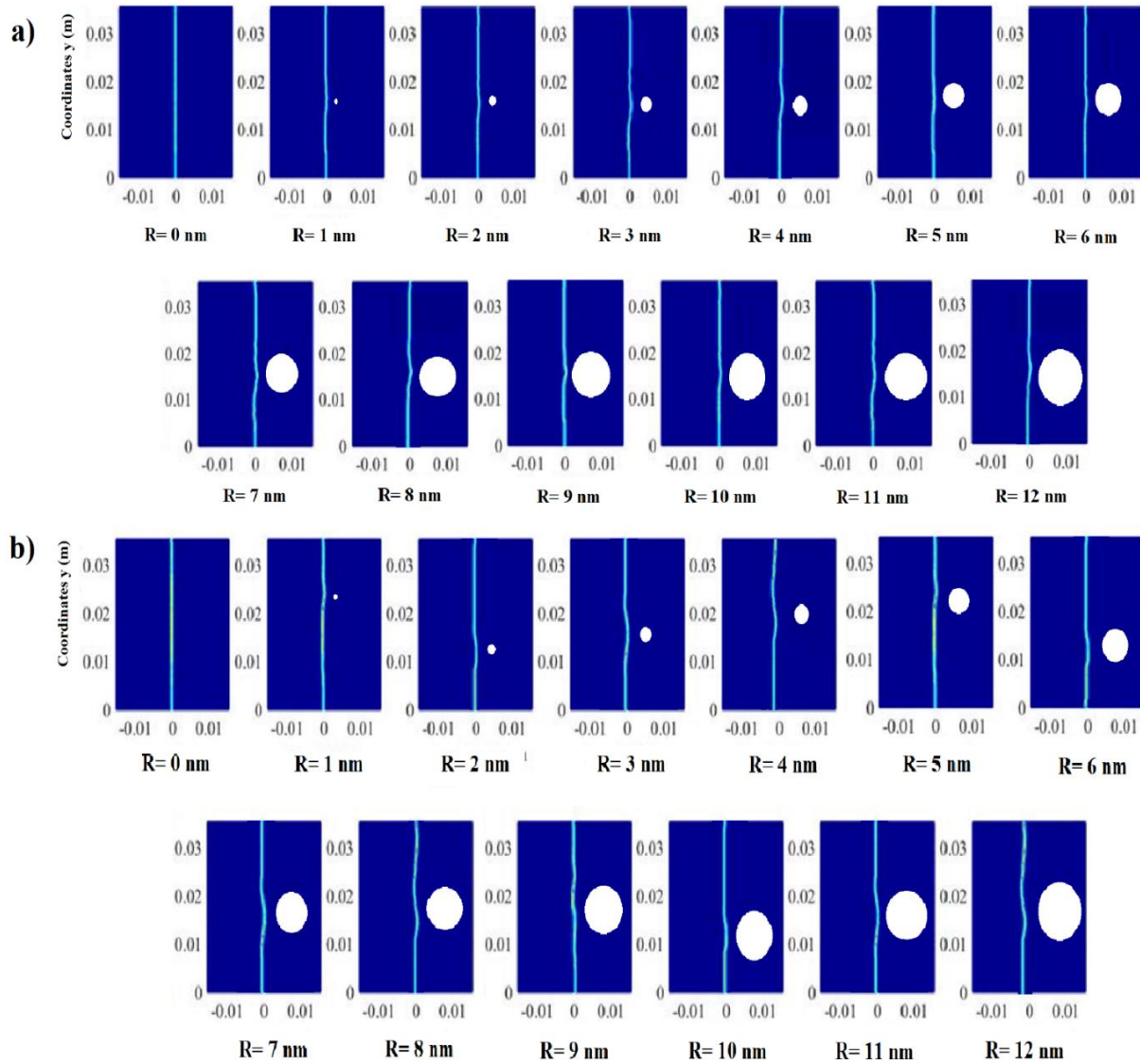
Discretized with  $1.6 \times 10^8$  material points spaced uniformly at  $\Delta x = 6 \times 10^{-9}$  m, the PD models have a defined horizon size of  $\delta = 3.018 \Delta x$ . Employing a consistent time step size of  $\Delta t = 0.081 \mu\text{s}$ , the simulation includes a critical stretch of  $Sc = 0.01072$ , corresponding to an energy release rate of  $G_0 = 220 \text{ J/m}^2$ . The crack propagation behavior under varied loading conditions is depicted in Figure 17 as analyzed through PD. When the structure is devoid of any porosity, the crack propagation follows a self-similar pattern. In contrast, with the presence of porosities within the structure, the crack path undergoes a noticeable shift and veers towards the porosities. This is consistent with the observed crack propagation pattern in the experimental tests [85].

In addition, larger holes result in a higher deflection of the crack. The crack path deviates

towards the defect upon its approach, returning to its original trajectory after propagating nearby. The crack trajectory appears curved and symmetric concerning the local axis of the porosity. Under both velocity boundary conditions, the crack advances towards the porosity, halts momentarily at its edge without intersecting, and subsequently proceeds towards the designated position of the applied boundary condition. To evaluate and calculate the crack propagation speed according to equation 29 [26]:

$$V = \frac{x_p - x_{p-1}}{t_p - t_{p-1}} \quad (29)$$

where the current crack tip position ( $t_p$ ) is indicated as  $x_p$ , while the previous crack tip position ( $t_{p-1}$ ) is denoted as  $x_{p-1}$ .



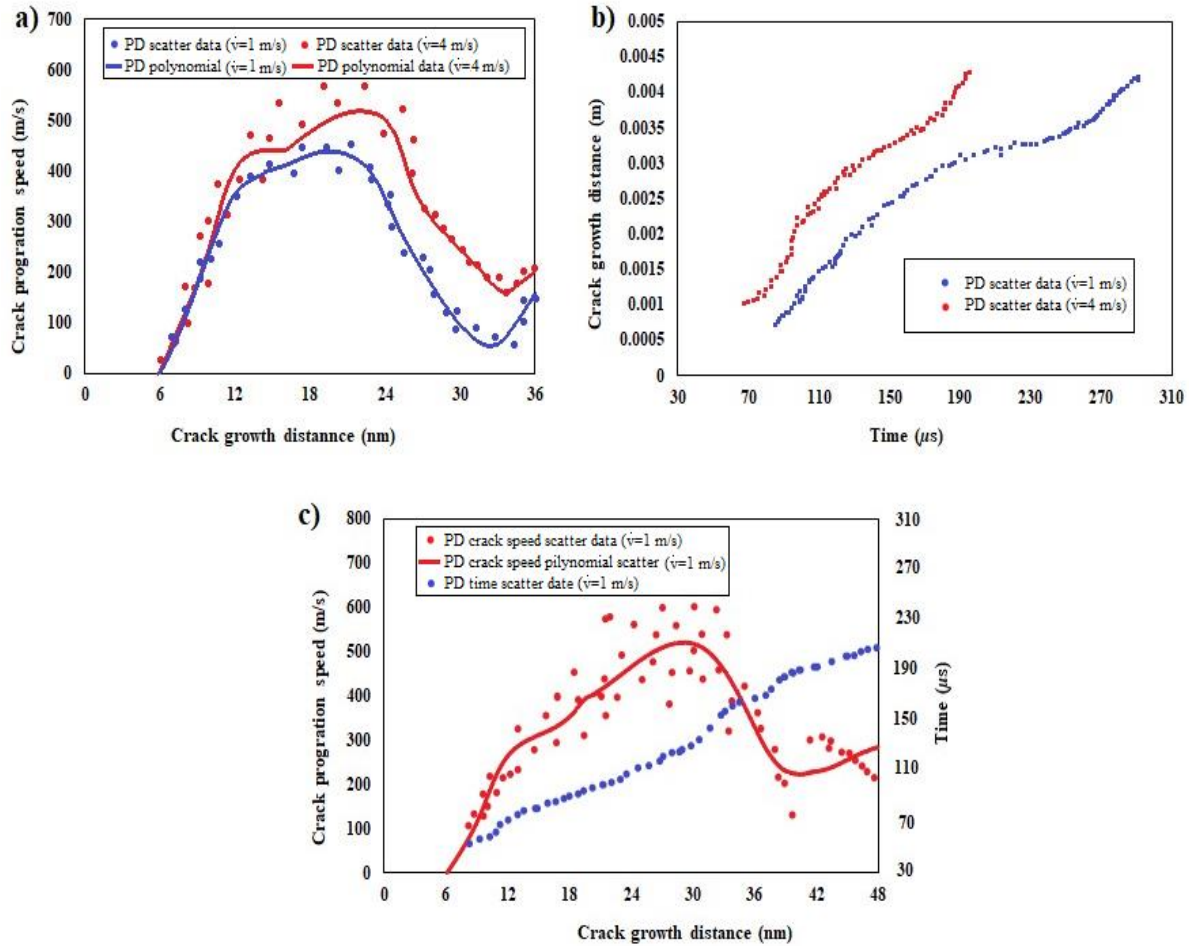
**Fig. 17.** PD Damage maps of 3Y-TZP+ x Al<sub>2</sub>O<sub>3</sub> dental plates with varying porosity radius R and under velocity boundary conditions: a)  $\dot{v} = 1$  m/s at  $t = 480 \mu\text{s}$ , b)  $\dot{v} = 4$  m/s at  $t = 280 \mu\text{s}$

In Figure 18-a, the diagram shows the relationship between crack propagation speed and crack growth. Initially set at  $\dot{v} = 1$  m/s, the speed increases to  $V = 400$  m/s as the crack tip nears a nanoporosity with a radius of  $R = 6 \times 10^{-9}$  m at coordinates  $x = 0$  m and  $y = (dy - 0.02)$  m. As the crack advances towards the nanoporosity, its propagation speed diminishes gradually, hitting a low point at  $V = 200$  m/s when the crack tip is positioned at  $x = 6 \times 10^{-4}$  m and  $y = dy + 0.025$  m. Subsequently, there is a brief pause in the crack propagation, lasting about  $10 \mu\text{s}$ , before the crack resumes its original course with an accelerating speed, as illustrated in Figure 18-b. The trend remains consistent with a higher velocity BC of  $\dot{v} = 4$  m/s, as depicted in Figure 18-a. In this scenario, the crack propagation speed escalates, peaking at  $V = 600$  m/s. As the crack approaches the porosity, it veers towards it while the speed gradually decreases, reaching  $V = 400$  m/s. The

crack also curves towards the porosity with a decreasing speed to  $V = 400$  m/s. After a very short crack arresting period of  $3 \mu\text{s}$  (Figure 18-b), the crack recovers and continues to propagate away from the porosity with an increasing speed. The phenomenon of crack arrest has been observed in multiple tests, and it is similar to the results obtained from PD simulations. However, detecting crack arrest is challenging as the arrest period is very brief. Furthermore, it is observed that the occurrence of crack arrest is dependent on the applied force, as shown in Figure 18-b. A lower velocity BC results in a longer delay in crack propagation. In addition, Figure 18-c illustrates the findings regarding crack propagation speed and time when applying a velocity boundary condition of  $\dot{v} = 1$  m/s to a plate containing a nanoporosity with a radius of  $R = 6 \times 10^{-9}$  m. Significantly, a clearly defined crack arrest phase, lasting approximately  $13 \mu\text{s}$ ,

initiates at  $t = 138 \mu\text{s}$  coinciding with the crack tip position at  $dy = 0.003 \text{ m}$ . Concurrently, the crack propagation speed decreases to  $V = 130 \text{ m/s}$  before returning to its original pace. A potential explanation for the more distinct crack arrest

phase could be the predominant linear propagation of the crack, with minimal deviation towards the porosity, as visualized in Figure 17-a for  $R = 6 \times 10^{-9} \text{ m}$ .



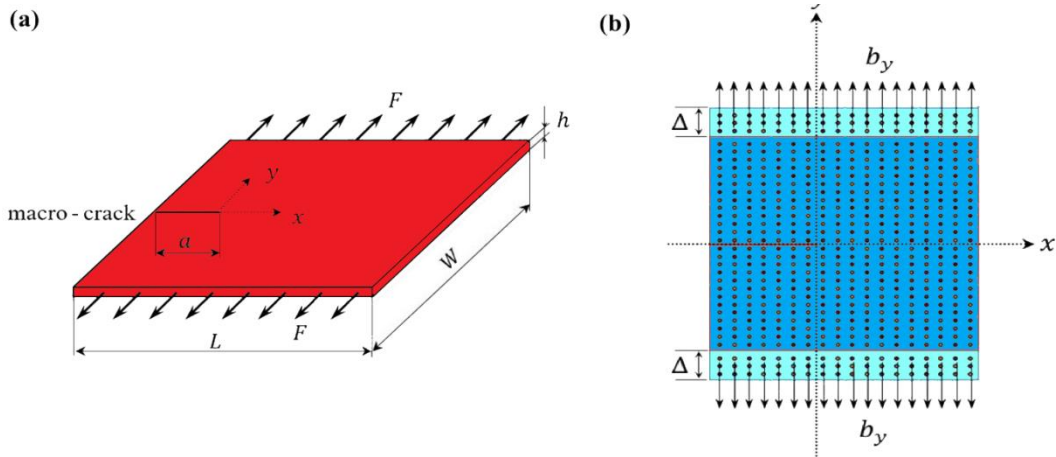
**Fig. 18.** Behavior of a dental plate ceramics sample with 3Y-TZP+ 0.5wt%  $\text{Al}_2\text{O}_3$ , porosity radius  $R = 12 \text{ nm}$ , under velocity boundary conditions of  $\dot{v} = 1 \text{ m/s}$  and  $\dot{v} = 4 \text{ m/s}$ . a) Crack propagation speed vs. growth distance, b) Crack length evolution over time, c) Crack propagation speed, time, and growth distance for  $R = 6 \text{ nm}$  under  $\dot{v} = 4 \text{ m/s}$

#### 4.4. Influence of Micro-cracks on Macro-crack Propagation in Stress-Loaded Dental Ceramic Plate

The geometric parameters influencing the interaction between macro- and micro-cracks include crack dimensions, distances between micro-cracks, and their orientation. In the PD model outlined in the study, a macro-crack with dimensions  $a = 0.055 \text{ m}$  is situated on the left side of the plate, as depicted in Figure 19-a. Furthermore, the model encompasses five distinct scenarios involving distributed micro-cracks, each with a length of  $a_c/a = 0.15$ .

The micro-cracks distances are represented by  $a/s$  and  $a/r$ , with  $s = r = 2$ . These micro-cracks are situated around and in front of the macro-crack tip, as shown in Figure 20. The configurations of the micro-cracks are analyzed

for cases (a) to (e), with the crack inclination angle  $\alpha$  varying from  $0^\circ$ ,  $45^\circ$ , to  $90^\circ$  in each scenario. The micro-cracks are positioned in a regular manner within the  $+y$  and/or  $-y$  domains and in front of the main crack tip. Geometric parameters for the 3Y-TZP/ $\text{Al}_2\text{O}_3$  plate consist of a length ( $L$ ) of  $0.2 \text{ m}$ , width ( $W$ ) of  $0.33 \text{ m}$ , and thickness ( $h$ ) of  $0.005 \text{ m}$ . The material properties include an elastic modulus of  $250 \text{ GPa}$  and  $\nu$  of  $0.3$ . Quasi-static PD simulations are carried out using an explicit time integration scheme with a stable time step size of  $\Delta t = 1$ , incorporating the adaptive relaxation technique [54]. During dynamic PD simulations, a time step size of  $\Delta t = 0.2 \mu\text{s}$  is utilized [53]. The stability criterion for the dental plate differs as it depends on the horizon size ( $\delta = 3.018 \Delta x$ ), with the spacing between points set at  $\Delta x = 12 \times 10^{-9} \text{ m}$  instead of the previously used  $\Delta x = 6 \times 10^{-9} \text{ m}$ .



**Fig. 19.** a) Dental plate with a macro-crack in quasi-static loading, b) Discretization of the dental plate with a macro-crack in body force density boundary conditions, following the model by Karpenko et al. [26]

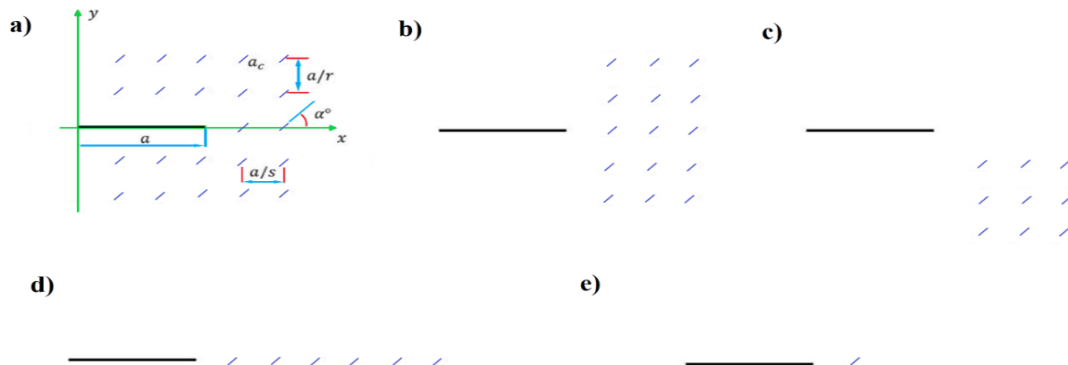
#### 4.5. Critical Load for Dental Plates with Micro-Cracks

The aim is to investigate how micro-cracks ahead of the macro-crack tip impact its propagation. Five different models of micro-crack distributions are studied, as displayed in Figure 20. The dental plate is under symmetric quasi-static compressive loading in the y-direction, as depicted in Figure 19-a. After conducting a convergence study, it was established that employing 3000 time steps with a time step size of  $\Delta t = 1$  s is effective for achieving quasi-static loading conditions using the adaptive dynamic relaxation method [29]. Initially, a load of  $F = 100$  N is applied, leading the system to a steady-state solution. Then, the applied force is incremented by  $\Delta F = 1$  N after every 3000 time steps. The evaluation involves assessing the elongation of bonds between material points. When the elongation surpasses the critical elongation threshold ( $s > s_c$ ), the applied force is recognized as the critical load  $p$ . These critical loads are labeled as  $p^0$  for a dental plate featuring a single macro-crack and  $p^*$  for the plate with both a macro-crack and distributed micro-cracks. The PD model is discretized using 118,520 material points, as illustrated in Figure 19-b. The material

points are uniformly spaced with a distance of  $\Delta x = 10 \times 10^{-9}$  m. Moreover, the horizon parameter  $\delta$  is established at 3.018 times the value of  $\Delta x$ . To replicate the loading scenario, a quasi-static loading condition is imposed through a body force density, as described in equation 30 [26].

$$b_y = \frac{F}{\Delta V_{\Delta}} \quad (30)$$

where the volume of the boundary layer is represented by  $\Delta V_{\Delta}$ , with a specific value set at  $\Delta V_{\Delta} = 1.98 \times 10^{-6}$  m<sup>3</sup>. The PD numerical models are under investigation to explore the connection between the SIF of the case featuring a macro-crack, labeled as  $K_0$ , and those scenarios involving micro-cracks, denoted as  $K^*$ . Throughout this analysis, a uniform fracture energy value of  $G_0 = 560$  J/m<sup>2</sup> is considered across all cases. The numerical model undergoes assessment using a consistent critical stretch-based criterion, where  $s_c^* = s_c = 0.0048$ , applied to all five instances of micro-crack distributions, as well as to the scenario with only one macro-crack featuring  $s_c^*$ . It is important to highlight that the critical stretch remains unaffected by the micro-crack layouts or the varying crack inclination angles  $\alpha$ , ranging from 0°, 45°, and 90°.

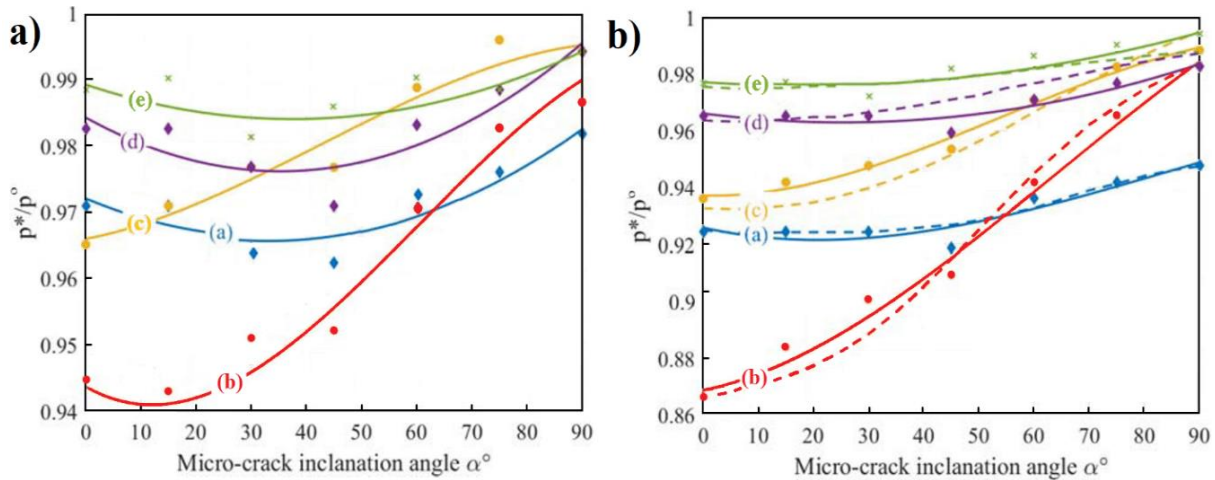


**Fig. 20.** Different arrangements of micro-crack distributions: a) Micro-cracks encircling the main crack tip, b) Micro-cracks positioned ahead of the crack tip, c) Uneven distribution of micro-cracks (+y domain) in front of the main crack tip, d) Cluster of micro-cracks located in advance of the crack tip, e) Solitary micro-crack positioned in front of the main crack tip

The PD simulations have been executed, with the outcomes depicted in Figure 21. The study involved assessing the ratios of critical loads,  $p^*$  and  $p^0$ . It was noted that the impact of micro-cracks on crack propagation aligns with the anticipated pattern from analytical solutions [34]. Nonetheless, the analytical findings revealed lower ratios of critical loads for inclination angles spanning from  $0^\circ$  to  $45^\circ$  when compared to the results derived from PD simulations. The discrepancy can be linked to the uniform critical stretch utilized in the PD simulations. Chen and Chu [86] offer insights suggesting that the SIF decreases when a multitude of micro-cracks encompass the primary crack.

Kachanov [87, 88] has shed light on the influence of micro-crack arrays and crack inclination angles on the structural geometry, resulting in either shielding effects or weakening phenomena. By exploring two distinct configurations of micro-crack distributions, namely collinear cracks and stacked cracks, He observed contrasting behaviors. Collinear cracks were associated with a stress amplification effect, leading to an increase in the SIF. In contrast, stacked configurations exhibited a stress

shielding effect, causing a reduction in the SIF. He studied how crack dimensions and distances affect amplification and shielding effects. Depending on the crack distribution, these effects may compete. For mode I traction BC, he noted that when distances between collinear and stacked cracks exceed the crack length, the shielding effect dominates. For an amplification effect, Kachanov recommends smaller distances between collinear cracks compared to stacked cracks [88]. The number and orientation of cracks also impact the SIF, with more parallel stack cracks leading to a decrease in SIF. In contrast, in the scenario of a coplanar row of cracks, the SIF escalates and nears a value of 1 for the ratio of critical loads ( $K^*/K_0$ ). Furthermore, the arrangement of micro-cracks surrounding the primary crack induces a stress-shielding effect. This observation has been supported by other researchers [89, 90], who have noted that arrays of micro-cracks can lead to SIF amplification at the macro-crack tip. Rotating the micro-cracks within the range of  $0^\circ$  to  $90^\circ$  (where  $90^\circ \geq \alpha > 0$ ) has a notable effect on the macro-crack. The arrangement of these rotated micro-cracks leads to an escalation in the SIF as the rotation angle increases from  $0^\circ$  to  $90^\circ$ .



**Fig. 21.** The critical load ratios for various micro-crack orientations a) Constant critical stretch for micro-crack distribution (points: PD scatter data, solid lines: PD polynomial) and b) Micro-crack distribution cases:  $K^*/K_0 =$  (a) 0.92, (b) 0.86, (c) 0.94, (d) 0.98, (e) 0.99 (Markers: PD scatter data, dashed lines: PD polynomials, solid lines: analytical solution for the same cases)

In the PD analysis, micro-crack distribution diagrams show that the spacing between micro-cracks is greater than the crack length ( $a/s \gg a_c$ ), indicating a prevalent shielding effect. Consequently, SIF in setups with micro-cracks is expected to be lower than in those with a single macro-crack, resulting in  $K^* < K_0$ . In the PD model, the effect of micro-cracks at a rotational angle  $\alpha$  of  $0^\circ$  on the SIF of the macro-crack tip is considered using analytical solutions from reference [88]. The designated relationships for  $K^*/K_0$  in the PD model are (a) 0.92, (b) 0.86, (c) 0.94, (d) 0.98, and (e) 0.99.

The numerical model's  $s_c^*$  values are adjusted by incorporating the selected  $K^*/K_0$  ratios to keep the calculated values below the original  $s_c$  values. Additionally, to address the impact of micro-crack orientations on the macro-crack tip, the critical stretch ratios are incrementally elevated. When the rotation angle  $\alpha$  is  $90^\circ$ , the resulting values are approximately 0.0034 for cases (a)-(c) and 0.0036 for cases (d)-(e). The variation in critical shrink values among these cases stems from the positioning of the micro-cracks. Specifically, when the micro-cracks are located

behind and above the macro-crack tip at a rotation angle of 90°, the  $K^*/K_0$  ratio drops below 0.96 [88]. Integrating the  $K^*/K_0$  relationship into the PD model yields more accurate results compared to the analytical solution [34]. This observation provides additional confirmation of the significant role played by crack propagation with respect to both the distribution of micro-cracks and the deviation angle. It underscores the intricate relationship between these factors in influencing the structural integrity and behavior of the material under consideration.

#### 4.6. Micro-crack Influence on Fracture Propagation in Dental Plates

To determine the crack propagation velocities, dynamic boundary conditions are applied to the dental plate illustrated in Figure 22. The plate undergoes symmetric loading with velocity constraints of  $v^* = 0.4$  m/s enforced on the plate edges at  $y = W/2$  and  $y = -W/2$ . The boundary conditions detailed are implemented on the fictitious boundary layer  $R_i$ , matching the horizon size ( $\delta = 3.015 \Delta x$ ). These boundary conditions are enforced in accordance with equations 31-34 [91]:

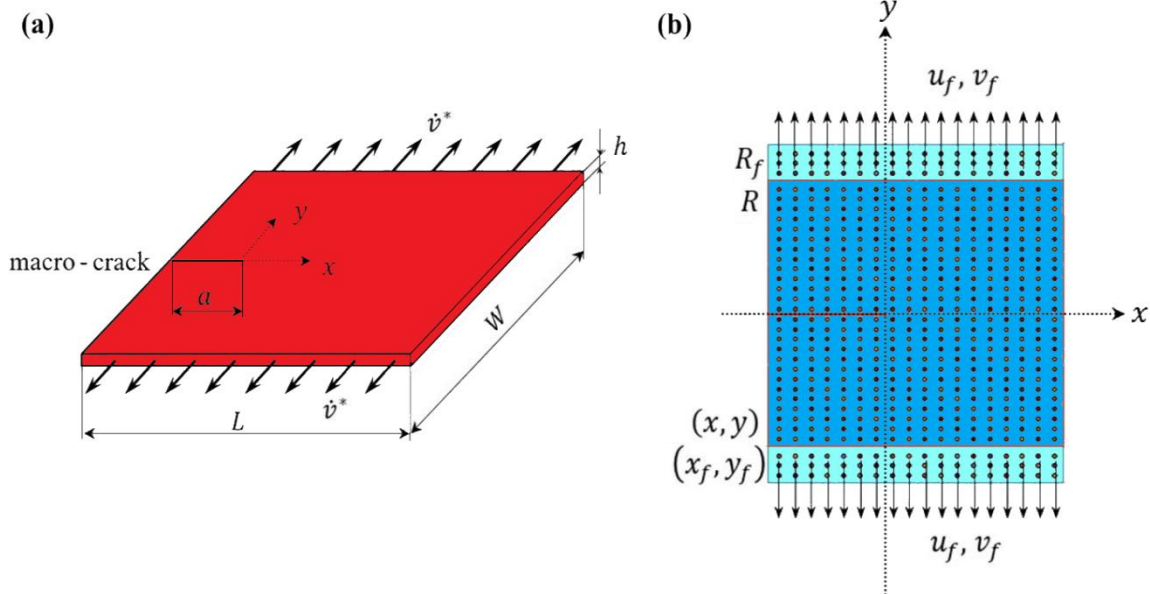
$$u_f(x_f, y_f, t + \Delta t) = u(x, y, t) \quad y_f \in R_{f(y=\frac{W}{2})} \quad (31)$$

$$v_f(x_f, y_f, t + \Delta t) = 2v^* \left( x, y = \frac{W}{2}, t + \Delta t \right) \times t - v(x, y, t) \quad (32)$$

$$u_f(x_f, y_f, t + \Delta t) = u(x, y, t) \quad y_f \in R_{f(y=\frac{W}{2})} \quad (33)$$

$$v_f(x_f, y_f, t + \Delta t) = 2v^* \left( x, y = -\frac{W}{2}, t + \Delta t \right) \times t - v(x, y, t) \quad (34)$$

The PD model in Figure 22-b is discretized into 272,800 material points with a uniform spacing of  $\Delta x = 6 \times 10^{-9}$  m. Simulations run with a time step of  $\Delta t = 0.2 \mu s$  and end at  $t = 400 \mu s$ . A constant fracture energy model is used, where the critical fracture energy remains consistent regardless of crack propagation speed [92]. The PD numerical models for scenarios (a) to (e) depicted in Figure 20 are first investigated by analyzing how the SIF of the macro-crack case,  $K_0$ , relates to the cases with micro-cracks,  $K^*$ . This study is conducted under consistent critical stretch conditions. Subsequently, the PD model delves into the correlation between  $K^*/K_0$ , where  $K^*$  denotes the SIF of cases featuring micro-cracks, and  $K_0$  represents the SIF of the macro-crack case. The values of  $K^*/K_0$  vary for each case as follows:  $K^*/K_0 =$  (a) 0.92, (b) 0.86, (c) 0.94, (d) 0.98, and (e) 0.99. The critical stretch values are adjusted by multiplying them with the corresponding  $K^*/K_0$  ratios to implement these relationships.

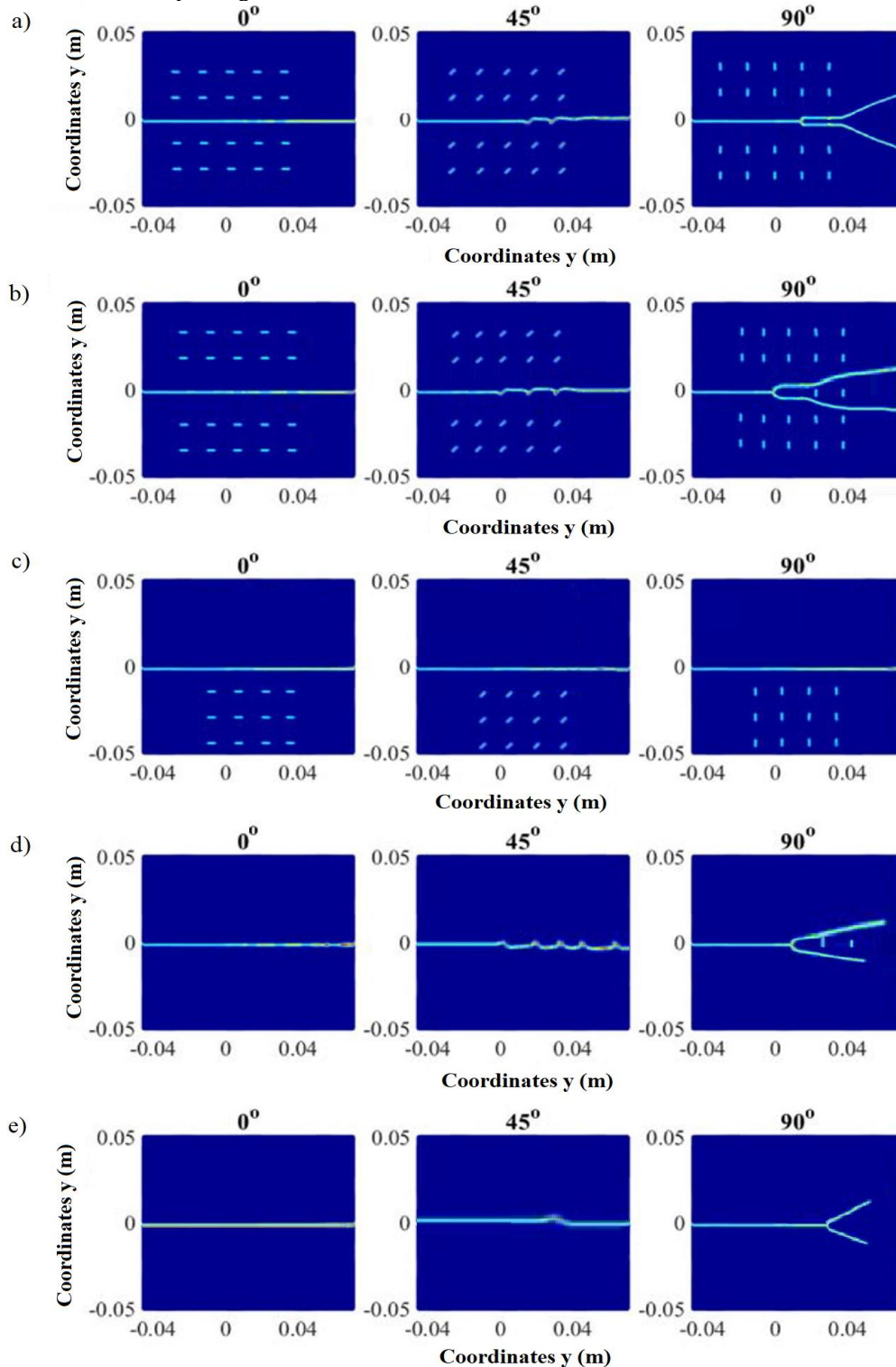


**Fig. 22.** a) Depiction of a square dental plate with a macro-crack under velocity boundary conditions. b) Discrete spatial representation of the square dental plate featuring a side macro-crack under prescribed boundary conditions, following the model by Karpenko et al. [26]

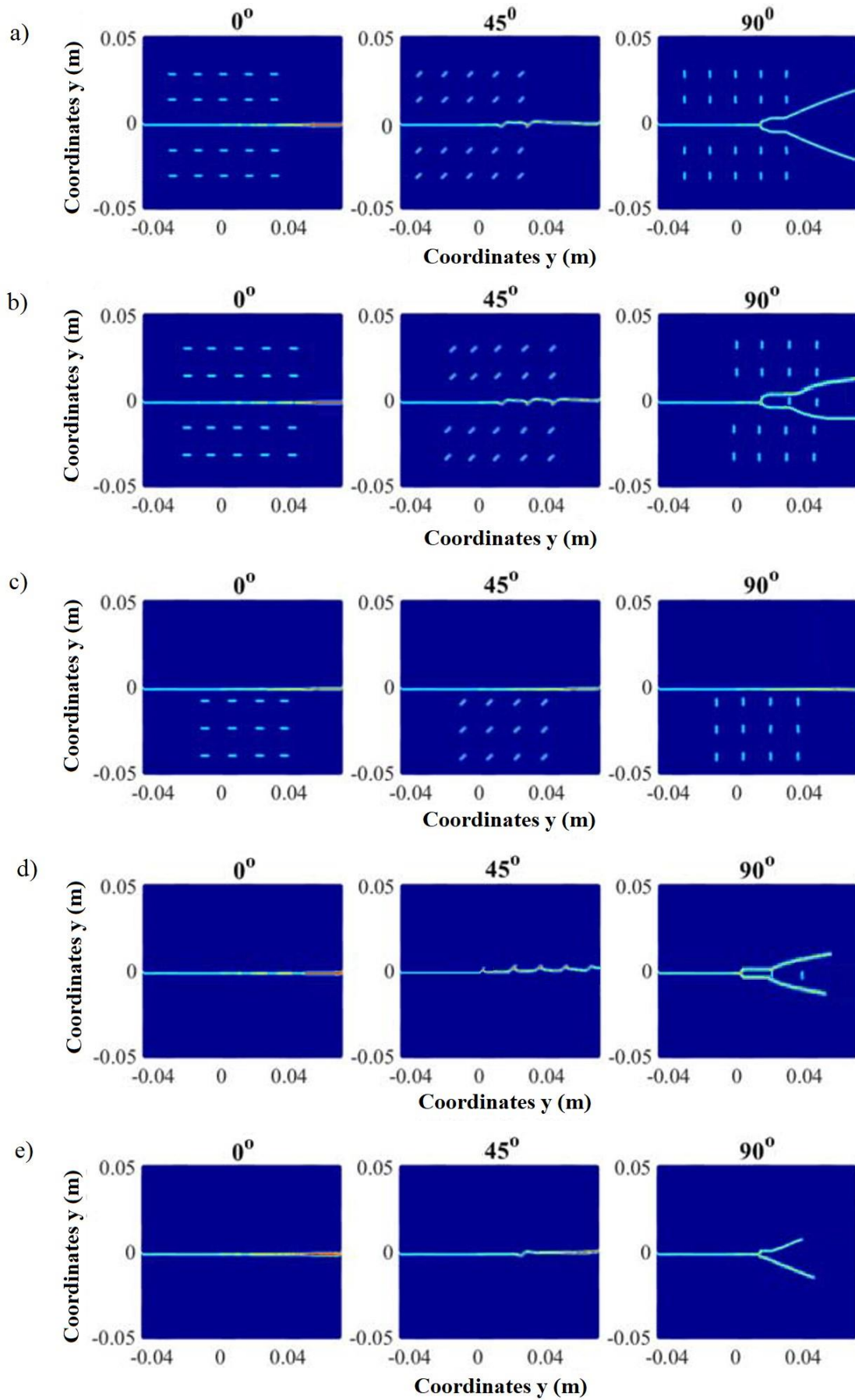
In Figure 23, the damage plots for all cases are displayed under a fixed critical stretch condition.

Figure 24 presents plots for cases with  $K^*/K_0 \neq 1$  and varying micro-crack inclination angles ( $\alpha = 0^\circ, 45^\circ$ , and  $90^\circ$ ). Interestingly, the orientation of the micro-cracks, represented by the inclination angles, influences the macro-crack propagation behavior, showing a tendency for the macro-cracks to follow the orientation of the micro-cracks. Moreover, placing the micro-crack

at the leading edge perpendicular to the macro-crack ( $\alpha = 90^\circ$ ) results in the bifurcation of the macro-crack. The crack propagation patterns observed in both critical stretch value setups are notably similar. The PD simulation outcomes highlight the significant impact of the micro-crack types, their sizes, spatial arrangements, and orientations on the toughening mechanisms operating within the system.



**Fig. 23.** Evolution of damage resulting from crack propagation in the 3Y-TZP+ 0.025 wt%  $\text{Al}_2\text{O}_3$  sample, with a consistent critical stretch across scenarios a to e, showcasing multiple micro-cracks oriented at angles of  $\alpha = 0^\circ, 45^\circ$ , and  $90^\circ$



**Fig. 24.** Progression of damage from cases a to e, where  $K^*/K_0$  represents the micro-crack distribution:  $K^*/K_0$  = a) 0.92, b) 0.86, c) 0.94, d) 0.98, e) 0.99, with micro-cracks oriented at angles of  $\alpha = 0^\circ$ ,  $45^\circ$ , and  $90^\circ$  during crack propagation in the 3Y-TZP + 0.025 wt%  $\text{Al}_2\text{O}_3$  sample

## 5. Conclusions

In the current study, a series of experiments were conducted to fabricate dental plate ceramics using a combination of 3Y-TZP + x wt%  $\text{Al}_2\text{O}_3$ . The research delved into exploring various aspects such as mechanical properties, resilience to aging, and the application of ordinary state-based peridynamics models to address complex issues associated with nanoporosities and interactions between micro and macro-cracks. Summary of the major discoveries and insights derived from this study:

- 1) The specimens displayed a uniform microstructure with increased density and a grain size falling within the range of 80 nm to around 100 nm, attributed to the low-level doping of  $\text{Al}_2\text{O}_3$ .
- 2) By increasing the amount of  $\text{Al}_2\text{O}_3$  additive up to 0.5 wt% in 3Y-TZP dental ceramics, the values of relative density, hardness, Young's modulus, fracture toughness, and flexural strength increased to 99.5%, 15.1 GPa, 280 GPa, 9.97  $\text{MPa}\cdot\text{m}^{1/2}$ , and 355 MPa, respectively.
- 3) The dental ceramics exhibited high hardness and significant fracture toughness, primarily attributed to the mechanism of phase transformation toughening.
- 4) Enhanced resistance to aging and superior mechanical properties were attained through the incorporation of 0.25 wt%  $\text{Al}_2\text{O}_3$  doping.
- 5) Nanoporosities were found to play a critical role in structural behavior and crack propagation, as evidenced by the PD numerical model showcasing crack arrest phenomena and deviations in trajectory towards the porosities.
- 6) The parameter  $K^*/K_0$  significantly impacted results, with close agreement between updated  $K^*/K_0$  values and analytical solutions for different micro-crack distributions, indicating facilitation of crack propagation with micro-crack allocation.
- 7) Analysis of crack propagation speeds revealed a shielding effect with microcracks ahead of the macro-crack, reducing crack speeds by 15%.

## Nomenclature

$\Delta V_d$  The volume of the boundary layer  
 $\alpha$  notched depth

$\theta$	Dilatation between material points in PD
$\rho$	Density
$\delta$	Horizon size in PD
$\kappa$	Bulk modulus in PD
$\mu$	Shear modulus in PD
$\nu$	Poisson's ratio
2-D	2-dimensional
3-D	3-dimensional
$\text{Al}_2\text{O}_3$	Alumina
$a/s$	Micro-cracks distance
$B$	Width of the specimen bar
BC	Boundary condition
$b$	Prescribed body force density field in PD
c- $\text{ZrO}_2$	Cubic Zirconia
CaO	Calcium oxide
$\text{CeO}_2$	Ceric oxide
$dp/dh$	Material stiffness
$E$	Young's modulus
$\text{Er}_2\text{O}_3$	Erbium oxide
FEM	Finite element method
FESEM	Field emission scanning electron microscopy
$G_c$	Critical energy release rate
$f_m$	Volume monoclinic fraction
$H$	A neighborhood of x
$h$	Thickness parameter in PD
$H_{x(i)}$	Interaction between material point $i$ and $j$ within its horizon
$H_v$	Vickers hardness
$K^*/K_0$	The ratio of the critical
$K_{IC}$	Fracture toughness in mode I
LTD	Low-temperature degradation

$l$	Support span
MAJ	Mehl-Avrami-Johnson equation
MgO	Magnesium oxide
MOR	Flexural strength
m-ZrO <sub>2</sub>	Monoclinic zirconia
$P_{\max}$	Applied load at fracture
$p$	Critical load
$p^*$	Critical load with a macro-crack and micro-cracks
PD	Peridynamics
PSZ	Partially stabilized zirconia
SELNB	Single-edge laser-notch beam
SIF	Stress Intensity Factor
$S_c$	Critical stretch
$s$	Distance between material in PD
TPB	Three-point bending
$t$	A pairwise force function
$t_p$	Current crack tip position
$t_{p-1}$	Previous crack tip position
t-ZrO <sub>2</sub>	Tetragonal zirconia
$u$	Displacement vector field
$\ddot{u}_{(i)}$	The acceleration of material point $i$ at time $t$
$V$	Velocity
$V_m$	Volume monoclinic phase
$V_{ms}$	Saturation of monoclinic phase
$W$	The thickness of the specimen bar
XRD	X-Ray diffraction
XFEM	Extended finite element method
$X_m$	Proportion of the monoclinic phase relative
Y <sub>2</sub> O <sub>3</sub>	Yttria
YSZ	Yttria-stabilized zirconia

$y$	Updated position of a material point in PD
$Y^*$	Stress intensity shape factor
ZrO <sub>2</sub>	Zirconia

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## Conflicts of Interest

The author declares that there is no conflict of interest regarding the publication of this article.

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