

*Research Article*

Annealed-Induced Recrystallization and Phase Evolution in Indented Magnesia-Stabilized Zirconia Ceramics

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Abstract: Ceramic components are often subjected to mechanical loads and high temperatures during operation, which can lead to the accumulation of residual stresses, formation of various types of defects, and initiation of phase transformations. Although the individual effects of mechanical stresses or high temperatures on ceramic materials have been extensively studied, the material's behavior under their sequential application, for example, during the annealing of previously deformed (indented) regions, remains of particular interest. The repeated annealing of ceramics can induce recrystallization processes, whose characteristics can differ significantly in previously stressed regions compared with undamaged regions of the material owing to the increased defect density and high concentration of local stresses. Partially stabilized Mg-doped ZrO₂ ceramics have attracted considerable attention owing to their complex, composite-like structure, in which tetragonal grains are embedded in a cubic matrix. This microstructure provides high mechanical strength to the ceramics; however, this multiphase structure may be less stable under mechanical loading and heating. The influence of residual stresses and defects on recrystallization during annealing should be studied. In this study, Raman spectroscopy was used to investigate local phase evolution in indentation sites following recrystallization induced by annealing. Indentation was found to cause only a slight decrease in the fraction of the tetragonal ZrO₂ phase and localized structural disorder at the center of the indentation site, where the stress concentration is highest. After annealing at 1000°C, the tetragonal phase fraction returned to its initial value. In contrast, annealing at 1100°C and 1200°C promoted the transformation of the cubic phase into the monoclinic phase and significant grain growth. This result indicates a metastable phase state after sintering and highlights the importance of assessing the phase stability of OCs under additional thermal and mechanical stresses.

Keywords: Indentation; Magnesia stabilized zirconia; Phase transformations; Raman piezo-spectroscopy; Recrystallization

1. Introduction

Ceramic materials are widely used in various industries due to their heat resistance, chemical inertness, and strength (Drazin et al., 2020; Edalati et al., 2025; Mahajan & Johnson, 2020; Shakirzyanov et al., 2024; Tokita, 2021). Ceramic materials can accumulate plastic deformation and residual stresses under mechanical loading, such as pressure or friction, despite their hardness and brittleness (Grigoriev et al., 2023; Zhou et al., 2024). This behavior is also reflected in cavitation erosion, where localized pseudoplastic deformation pits can form on ceramic surfaces. Their formation strongly depends on fracture toughness: ceramics with higher fracture toughness are less prone to pit formation because crack initiation under impact loading is suppressed (Fatjó et al., 2011). Mechanical stresses also lead to the formation of defects in the material (Ayode-Otitoju et al., 2020). In localized regions where such defects are concen-

trated, undesirable phenomena such as crack initiation can occur. These cracks may propagate over considerable distances from the microscopic scale, eventually leading to the failure of the entire ceramic component. Consequently, the mechanisms of crack nucleation and propagation during indentation have received considerable attention (Li et al., 2020). Cracks also serve as indicators of the stress state in ceramics. Recently, a Vickers indentation-based approach has been developed to estimate residual stresses by correlating the indentation size and crack length emanating from the indentation (Shan et al., 2024). Furthermore, phase transformations can occur in mechanically damaged regions (Garanin et al., 2025; Kariminejad et al., 2025).

These mechanically induced changes can significantly affect the properties of the material and make it less reliable. Such damaged material is often subjected to annealing to relieve residual stresses and restore its microstructure (Kayukawa et al., 2023; Yanaseko et al., 2024). The annealing of ceramics can lead to recrystallization, i.e., grain growth, as well as the growth of unstressed grains at defects and stress concentration sites (Karipbayev et al., 2025; Volodina & Shakirzyanov, 2025). During annealing, grain growth and enhanced diffusion promote microstructural recovery, leading to crack closure and healing. Therefore, it is important to study how annealing affects the morphology, phase composition, and microstructure of ceramics previously subjected to mechanical loading, as these characteristics may change significantly during heat treatment (Pangilinan et al., 2018; Rösler et al., 2007; Tan et al., 2020; Wang et al., 2023). This will lead to a better understanding of recrystallization processes, which is crucial for predicting ceramic material behavior under service conditions. If stress occurs during operation and the ceramic properties change, this can lead to brittleness. Thermal exposure of such ceramics can lead to phase transformations and morphological changes, and monitoring these processes is important (Frangulyan et al., 2018).

Currently, ZrO₂-based ceramics stabilized with various compounds are widely used. Y-stabilized ZrO₂ has attracted considerable attention because its metastable tetragonal phase provides excellent mechanical strength, fracture toughness, and reliability (Zhang et al., 2013). However, depending on the specific application, other dopants may be more suitable. Magnesium oxide can be a good choice in this case because of its large availability and significantly lower cost than yttrium oxide (Berar et al., 2020; Yuan et al., 2020). In addition, MgO is less susceptible to low-temperature degradation in the presence of moisture, which is typical for Y-TZP ceramics (Burger & Kiefer, 2021).

An interesting approach to partial stabilization using MgO was explored. MgO was used at a concentration of 3.2 wt.%, sintering was performed at 1750°C, and the system was rapidly cooled to 800°C. Annealing at 1750°C implies that this process occurs in the phase diagram region where only a cubic solid solution exists. The material passes through a region where the tetragonal and cubic phases coexist in a solid solution during cooling. Thus, a microstructure consisting of large cubic grains with fine tetragonal precipitates is formed during cooling. These tetragonal precipitates can be formed either by controlling the cooling rate or through appropriate post-sintering heat treatment. Cubic grains typically range in size from 30 to 70 µm, whereas the size of the tetragonal precipitates is limited to below 0.2 µm, which is necessary to retain the metastable tetragonal phase. If these tetragonal precipitates grow beyond the critical size, an immediate phase transition to the monoclinic phase occurs at room temperature. Thus, the size of the tetragonal precipitates must remain below the critical size to maintain the phase stability of the material. Conversely, the closer the size of the tetragonal precipitates is to the critical size (without exceeding it), the higher the mechanical strength and fracture toughness. These tetragonal precipitates are responsible for the excellent mechanical properties of this ceramic (Rainforth, 2004). This microstructure, comprising cubic grains with embedded tetragonal precipitates, is characteristic of ceramics subjected to classical transformation.

Compared to stabilized cubic zirconia, which is very brittle and has a mechanical strength of only about 200–250 MPa, partially stabilized Mg-doped zirconia exhibits a strength of 500–750 MPa (Swab et al., 2025). However, when the size of tetragonal precipitates is close to critical, the material can only be used at room temperature. For applications at elevated temperatures

of $\sim 800^{\circ}\text{C}$, the tetragonal precipitates must be smaller to maintain phase stability, resulting in a reduced mechanical strength of approximately 500 MPa (Lakshya et al., 2024).

The lenticular microstructure of partially stabilized MgO zirconia ceramics arises from heat treatment, which induces the formation and growth of tetragonal precipitates in the initially cubic matrix. This transformation from the cubic to the tetragonal phase results in residual compressive stresses in the tetragonal phase (Rajendran et al., 2020).

The processing of Mg-PSZ also requires tight control of the cooling rates and impurity levels (e.g., silica impurities can react with MgO to form forsterite, thereby reducing the dopant's effectiveness) (Burger & Kiefer, 2021). If Mg-PSZ is exposed to temperatures of approximately 1100°C – 1200°C for prolonged periods, some of the beneficial tetragonal precipitates may transform into the monoclinic phase or dissolve into the cubic matrix, leading to a reduction in toughness and strength. Prolonged thermal exposure (over-aging) can significantly degrade the room-temperature toughness of Mg-PSZ. This limitation can be mitigated by co-doping (Mg+Y) or by using the material within its recommended temperature range, but it remains an important consideration for long-term high-temperature applications (Burger & Kiefer, 2021).

The stabilization mechanism of MgO is closely related to its state in the zirconia system. When MgO is introduced into the zirconia system, it may dissolve into the zirconia lattice to form a solid solution or segregate to particle surfaces and grain boundaries (Castro et al., 2008). The state of MgO in the zirconia system would change the surface and bulk energy, thereby affecting the polymorphic stability. Consequently, both solid-solution formation and surface segregation play key roles in the phase stabilization of zirconia (Yuan et al., 2020).

This study examines the recrystallization of pre-indented ZrO_2 -based ceramics doped with MgO. The ceramics used in this study were produced using standard ceramic technology. Raman spectroscopy studies of phase transformations occurring in localized indented regions were particularly important. By combining localized mechanical loading with subsequent annealing, this study provides insight into the evolution of the phase composition and microstructure during recrystallization. Such studies are valuable for understanding the structural stability of zirconia ceramics and for assessing their reliability under combined mechanical and thermal effects, thereby preventing premature failure during service.

2. Methods

2.1 Sample Fabrication

The samples were prepared using zirconium oxide as the base material and magnesium oxide as a stabilizing additive (Sigma Aldrich, St. Louis, USA). The MgO concentration was 10 wt.%, which was the concentration at which partially stabilized zirconium ceramics were previously obtained (Borgekov et al., 2025). The powders were mixed in a Fritsch Pulverisette 6 planetary mill (Fritsch, Idar-Oberstein, Germany) for 30 min at a rotation speed of 250 rpm using a tungsten carbide grinding set. Polyvinyl alcohol (JSC EuroChem, Kovdor, Russia, 16/1 grade) solution was used as a plasticizer to form the green bodies. The oxide mixture was gradually added to a polyvinyl alcohol aqueous solution preheated to 90°C . A magnetic stirrer was used to obtain a homogeneous solution. The solution was dried in a drying oven for 5 hours at 60°C and then ground in a mortar. The polyvinyl alcohol content was approximately 1 wt.% relative to the dry powder mixture. Uniaxial pressing at 250 MPa using a hydraulic press was employed to form the samples. The resulting tablets were then sintered in air at 1700°C for 5 hours in a Nabertherm LHT 08/18 resistance furnace (Nabertherm, Lilienthal, Germany) at a heating rate of $10^{\circ}\text{C}/\text{min}$, followed by furnace cooling. Figure S1 (Supplementary Material) shows the overall sample synthesis procedure.

The samples were ground and polished before indentation. For ease of processing, the samples were embedded in epoxy resin and then ground using a Stress Tegramin-30 grinder (Struers, Ballerup, Denmark).

2.2 Sample Characterization

Indentation was used to introduce localized residual stresses and defects in a controlled and reproducible manner. After annealing, the indented regions were examined to evaluate recrystallization and phase evolution.

Raman spectroscopy was selected as the primary characterization technique in this study because it enables localized analysis of the phase composition. Unlike X-ray diffraction, where the diffraction peaks of the tetragonal and cubic ZrO_2 phases strongly overlap, Raman spectroscopy provides clearly distinguishable vibrational modes for these phases, allowing their reliable identification. A Raman spectroscopy setup (Spectr-M, Chernogolovka, Russia) equipped with a 532-nm laser was used in these studies. X-ray diffraction (XRD) was also employed as a complementary method to provide bulk phase analysis of the samples. XRD was performed on a Rigaku SmartLab diffractometer (Rigaku, Tokyo, Japan) equipped with a $\text{CuK}\alpha$ source. Sample indentation was performed using a Mellon DuroLine Vickers microhardness tester (Metkon Instruments Inc., Bursa, Turkey). The microstructure of the samples in the indentation imprint region was assessed immediately after indentation and after annealing for 5 min at 1000, 1100 and 1200°C using a Phenom ProX G6 scanning electron microscope (Thermo Fisher Scientific, Eindhoven, Netherlands). The heating rate was 10°C/min, followed by furnace cooling.

3. Results and Discussion

After polishing, the samples were examined by Raman spectroscopy. Figure 1a compares the MgO-doped samples before and after polishing. The Raman spectra of the unpolished sample indicate that it consists of tetragonal and cubic phases of ZrO_2 . The cubic phase is manifested by a broad background extending from 120 to 700 cm^{-1} , and the tetragonal phase through characteristic peaks at 147, 263, 319, 468, and 609 cm^{-1} (Materano et al., 2022). Raman bands at 826, 858, 884, and 969 cm^{-1} were detected, which are characteristic of the Mg_2SiO_4 phase. Mg_2SiO_4 phase may have formed on the ceramic surface as a result of SiO_2 contamination originating from the furnace heating elements during sintering. Lower intensities of the peaks of the tetragonal phase are observed after polishing. This change is primarily attributed to the mechanical effects of polishing, which may have introduced slight structural disorder and residual stresses in the surface layer, leading to broader and less intense Raman bands. The physical mechanisms underlying grinding-induced damage in ceramics are highly complex and strongly depend on the material microstructure and machining conditions (Xu et al., 2025). After polishing, the Raman peaks associated with the secondary Mg_2SiO_4 phase disappeared, indicating that this phase was present only on the ceramic surface (Kolesov & Geiger, 2004).

After polishing, the samples were indented using a microhardness tester with a force of 1 kg. The appearance of the indentation imprint as seen through an optical microscope is shown in Figure 1b's inset. The obtained result for the Mg-doped ZrO_2 ceramic was $\text{HV1} \sim 1200$. Cracks emanating from the mark's vertices formed in the indentation area, and material delamination was also observed. Figure 1b also shows the Raman spectra obtained from different locations within the indentation: the center, the vertex, and the midpoint of one side. The Raman spectra indicate that no significant changes in the phase composition occurred during the indentation of the material. Only a slight decrease in intensity and broadening of some peaks in the range of 250–550 cm^{-1} , related to the tetragonal phase, were observed for Raman spectra, collected at the indentation sites, which may be associated with disordering and residual stresses generated during indentation. In contrast, the Raman spectra recorded far from the indentation site did not exhibit this characteristic peak broadening, indicating the absence of significant stress-induced structural disorder.

To study the recrystallization processes in Mg- ZrO_2 ceramics, indented samples were annealed. Figure 2 shows the microstructure of Mg- ZrO_2 ceramics after annealing at 1000°C for 5 min. After annealing at 1000°C for 5 min, no significant changes in the morphology of the sample were observed, and no grain growth or recovery of the indentation mark was observed.

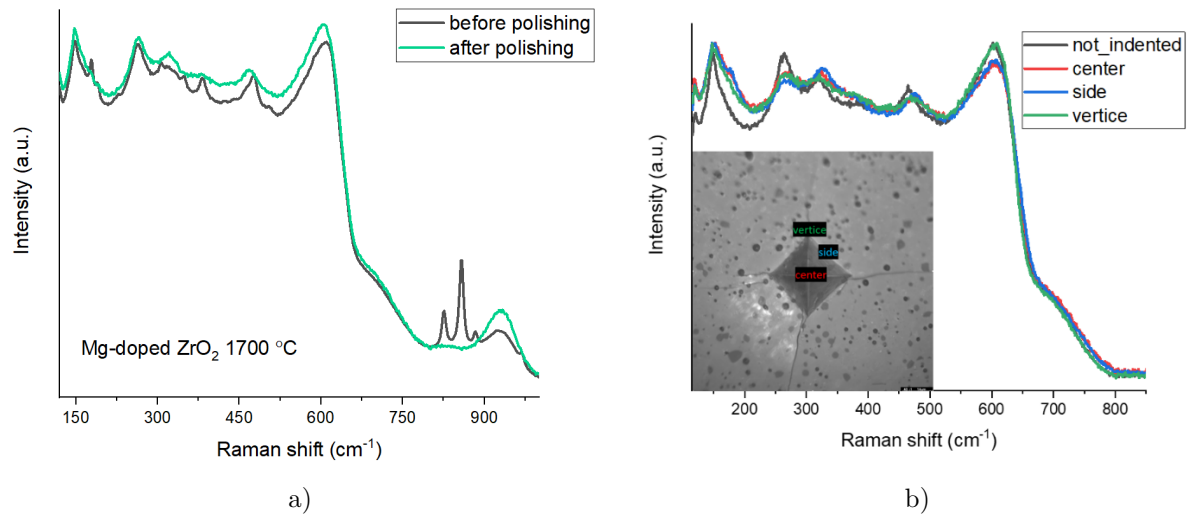


Figure 1 (a) Raman spectra of ZrO_2 ceramic samples before and after polishing with MgO .

(b) Raman spectra obtained at different locations within the indentation site: center, the center of the indentation; vertex, the corner of the indentation; and side, the midpoint of one side of the indentation, as shown in the inset of Figure 1a. The term “not indented” refers to the Raman spectra acquired from an unindented region located far from the indentation site

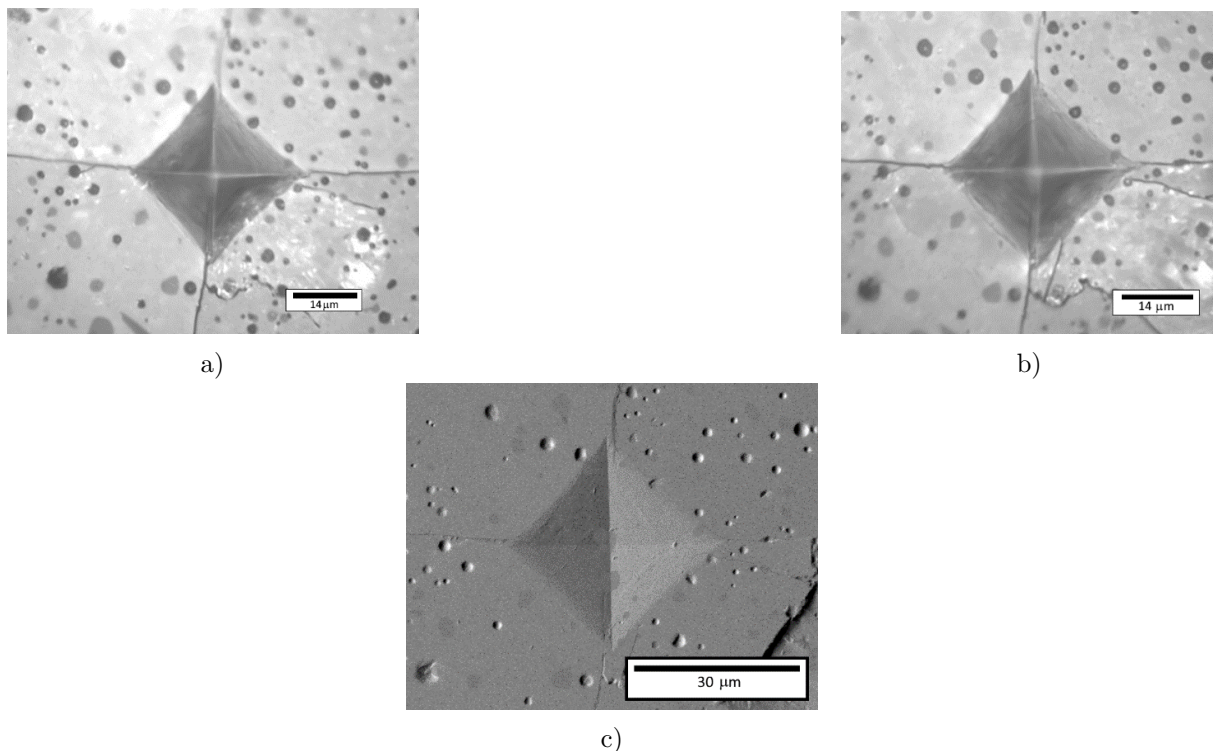


Figure 2 Optical images of indentation site before annealing (a), after annealing at 1000°C (b) and SEM image after annealing at 1000°C (c)

Figure 3 shows the Raman spectra obtained before and after annealing at the center of the indentation, where the stress concentration is highest, and in an unindented region far from the indentation, where indentation-induced residual stresses are absent, allowing the assessment of the influence of stress on recrystallization. Although the optical and SEM images showed no significant changes after annealing at 1000°C , changes in the spectra were detected in the indentation region after annealing. The figures show that the intensity of the peaks corresponding to the tetragonal phase of zirconium oxide increases after annealing. This observation may indicate the partial recovery of the tetragonal phase at the sample surface after annealing (Kambale et

al., 2019). Meanwhile, the spectra before and after annealing were almost identical in the region located far from the indentation center. The influence of residual stresses on the tendency to form a tetragonal phase during the heat treatment of the samples can be noted. A similar result for Ce-doped ZrO_2 was reported in a previous study, but the effect was more pronounced, which could be due to a higher fraction of the tetragonal phase and a lower fraction of the cubic phase (Volodina & Shakirzyanov, 2025).

Figure 4 shows the change in the microstructure of the samples after annealing at 1100°C . Although annealing at 1000°C did not result in any changes to the microstructure of the sample, increasing the annealing temperature to 1100°C resulted in significant changes, with recrystallization visible both within and outside the indentation region. The surface roughness increased significantly. As shown below, such a pronounced change in the microstructure may be related to a phase transition.

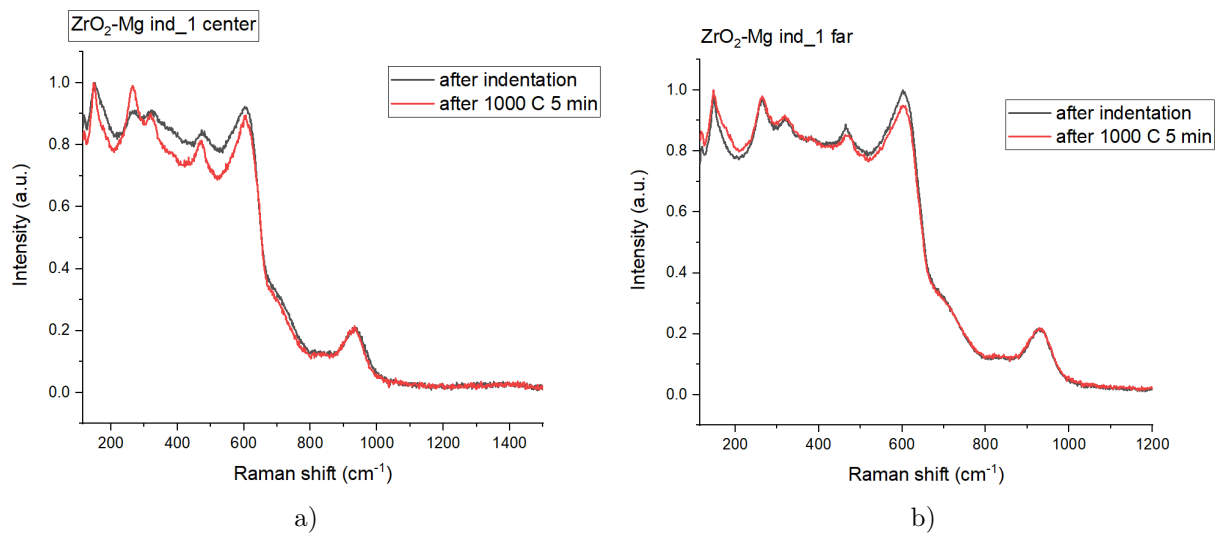


Figure 3 Comparison of Raman spectra at (a) the center of the indentation mark and (b) a significant distance from the indentation mark before and after annealing at a temperature of 1000°C

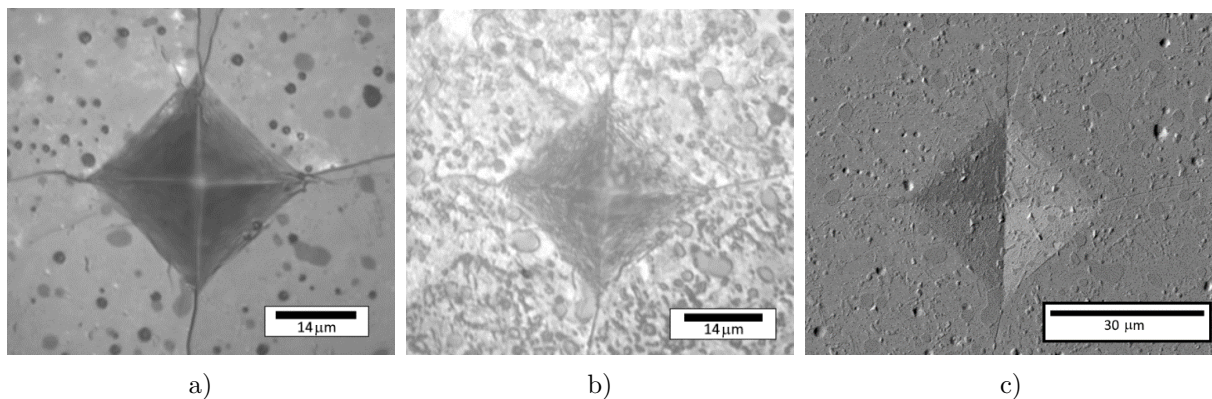


Figure 4 Optical images of indentation site before annealing (a), after annealing at 1100°C (b) and SEM image after annealing at 1100°C (c)

Figure 5 compares the Raman spectra recorded in and outside the indentation region before and after annealing. After annealing at 1100°C , a phase transition from cubic and tetragonal ZrO_2 to the monoclinic phase occurred. No significant difference was observed between the spectra at the indentation center and those far from the indentation mark because the entire sample transitioned to the monoclinic phase during annealing. This effect may be due to the instability of $\text{ZrO}_2\text{-MgO}$ solid solutions upon annealing and their tendency to undergo a phase transition to the monoclinic state. However, earlier studies of this system indicated that cubic

Mg-ZrO₂ solid solutions remained stable over a wide temperature range (Butz et al., 2009). A paper showed that the MgO-ZrO₂ solid solution could decompose into its constituent oxides at temperatures below 1400°C (Shakirzyanov et al., 2025), and additional studies also confirmed this assumption (Borgekov et al., 2025). In another study, hysteresis-like transitions of ZrO₂ from the monoclinic to the tetragonal phase were observed at temperatures below 1240°C (Mamivand et al., 2013). In addition, one of the studies on Mg-doped ZrO₂ indicated that partially-stabilized ZrO₂ usually represents a cubic matrix in which small particles of the tetragonal phase (<200 nm) are located, and if these particles increase to sizes larger than the critical one, this can provoke a transition to the monoclinic phase (Burger & Kiefer, 2021).

Interestingly, the sample did not fracture during the transition to the monoclinic phase, as often happens with ZrO₂ during phase transitions. The tetragonal phase is present as small inclusions within a more stable cubic matrix, which restrains volume expansion during the phase transition and thus prevents sample fracture.

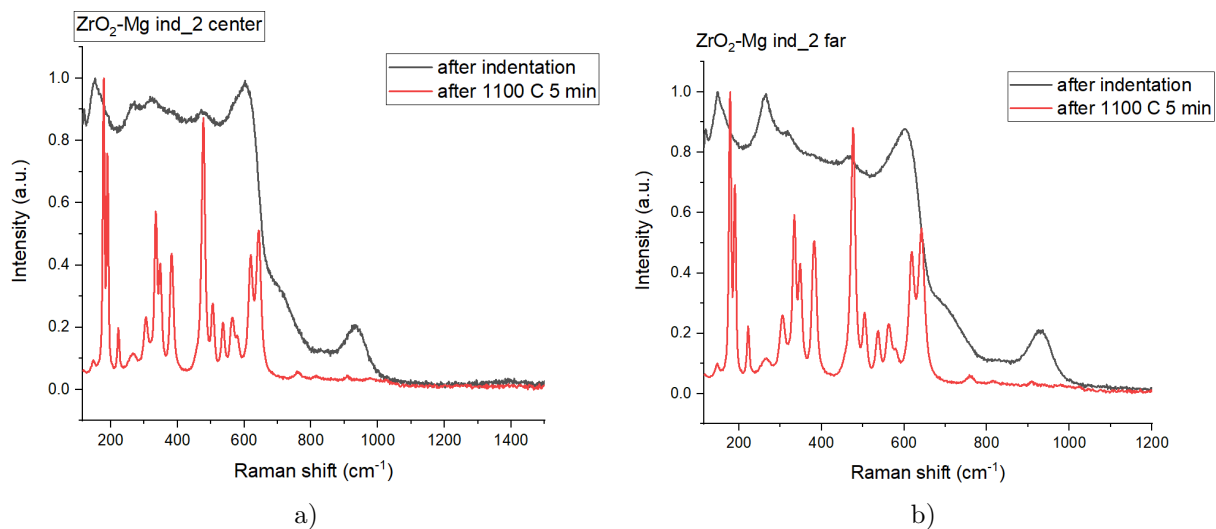


Figure 5 Comparison of Raman spectra at (a) the center of the indentation mark and (b) a significant distance from the indentation mark before and after annealing at a temperature of 1100°C

Furthermore, annealing was performed at 1200°C. Figure 6 shows the microstructural changes after annealing at 1200°C. Annealing at 1200°C resulted in more active grain growth than at 1100°C. The indentation mark was almost completely eliminated due to grain growth. Interestingly, dark regions formed along the indentation mark boundary, which most likely correspond to the areas most stressed during indentation.

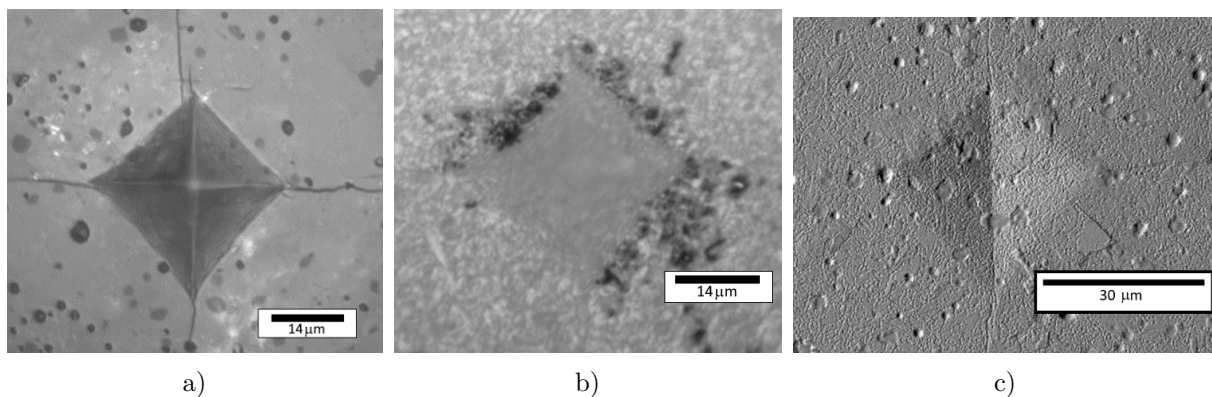


Figure 6 Indentation site before (a) and after annealing at 1200°C (b) after annealing at 1200°C SEM image (c)

Similar to annealing at 1100°C, annealing at 1200°C resulted in a phase transformation

from the cubic and tetragonal phases to the monoclinic phase. Figure 7 shows the Raman spectra of the ceramics before and after annealing, as well as at and away from the center of the indentation mark.

It was also important to evaluate the phase composition not only on the surface but also at some depth, for which XRD was used (Figure S2). The phase composition was obtained using Rietveld refinement on the Rigaku SmartLab software. R_{wp} was approximately 16 % for all samples. The crystal lattice parameters are listed in Table 1. Before annealing, the sample largely consisted of the cubic phase of ZrO_2 . Upon annealing at 1000°C, the phase composition of the sample remained virtually unchanged, although an increase in the intensity of the modes of the tetragonal phase of ZrO_2 was observed in the Raman spectra of the indented region. This confirms the local nature of the effect, indicating that it occurred only in a small region of the indentation zone and was caused by stresses and defects arising during indentation. Annealing at 1100°C caused a phase transition from the cubic phase to the monoclinic phase, with only 26% of the cubic phase remaining. Increasing the annealing temperature to 1200°C increased the content of the monoclinic phase to 90%. The XRD results are in good agreement with the Raman spectroscopy results. Figure 8 presents the final results of the study as a schematic representation of the change in the structure of the studied ceramics before and after annealing.

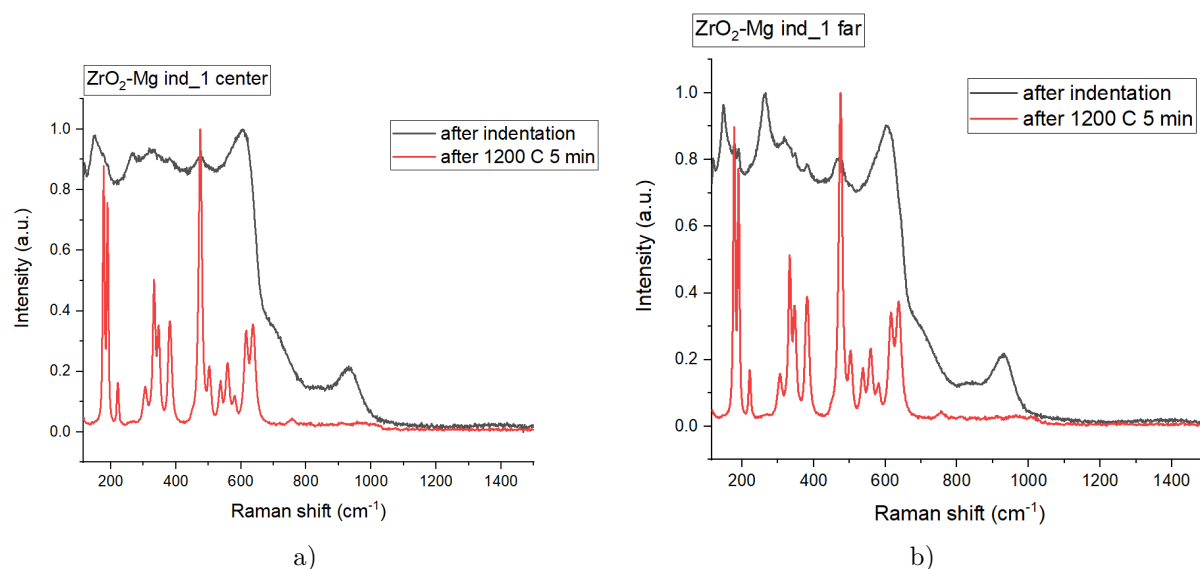


Figure 7 Comparison of Raman spectra (a) at the center of the indentation mark and (b) at a significant distance from the indentation mark before and after annealing at a temperature of 1200°C

A similar phase transition has also been reported in another paper, which states that if Mg-PSZ is subjected to prolonged annealing at temperatures of 1100–1200°C, some of the tetragonal grains may transition to the monoclinic phase or dissolve in the cubic phase of the matrix. One solution to this problem may be doping ZrO_2 with a mixture of MgO and Y_2O_3 oxides, which promotes better thermal stability (Lakshya et al., 2024; Rada et al., 2022).

Table 1 Crystal lattice parameters of samples under study

Sample	Cubic phase of zirconia	Tetragonal phase of zirconia		Monoclinic phase of zirconia				Periclase
	a , Å	a , Å	c , Å	a , Å	b , Å	c , Å	β , °	a , Å
Before annealing	5.074	3.070	6.394	—	—	—	—	4.217
1000 °C	5.073	3.587	5.062	—	—	—	—	4.216
1100 °C	5.085	—	—	5.154	5.192	5.335	99.105	4.219
1200 °C	—	3.624	5.075	5.153	5.207	5.320	99.169	4.217

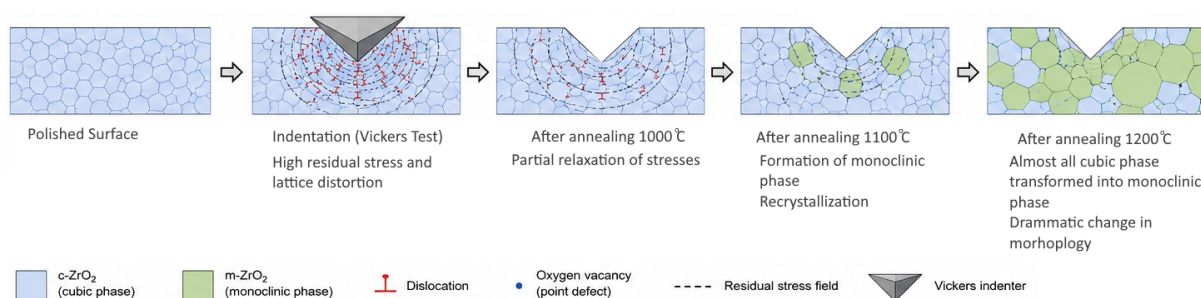


Figure 8 Schematic representation of the evolution of mechanical stresses in the ceramics under study

4. Conclusions

The indented regions of partially stabilized MgO-doped ZrO_2 ceramics were studied after recrystallization induced by annealing at 1000°C, 1100°C, and 1200°C. Annealing at 1000°C had little effect on the phase composition of the ceramics, except for a slight increase in the tetragonal phase content in regions deformed before annealing. However, annealing at 1100°C and 1200°C resulted in a phase transition in the ceramics, transforming ZrO_2 from a cubic-tetragonal phase to a monoclinic phase. This study demonstrated the influence of residual stresses in ceramics on grain growth processes, morphology changes, and phase composition changes during heat treatment. The obtained results emphasize the importance of a detailed analysis of the phase stability of doped zirconia ceramics under various external factors, including mechanical stresses and elevated temperatures. Understanding the mechanisms of phase transformations and the associated microstructural changes is essential for assessing the reliability and long-term performance of ceramic components under operating conditions. Such studies provide a basis for the informed selection of materials for critical engineering applications.

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Author Contributions

Conceptualization, Natalia Volodina, Dmitriy Shlimas and Artem Kozlovskiy; **methodology**, Natalia Volodina, Rafael Shakirzyanov and Yuriy Garanin; **software**, Malik Kaliyekperov, Natalia Volodina; **validation**, Natalia Volodina, Dmitriy Shlimas, Malik Kaliyekperov and Artem Kozlovskiy; **formal analysis**, Maxim Zdorovets; **investigation**, Natalia Volodina, Rafael Shakirzyanov, Yuriy Garanin, Artem Kozlovskiy; **resources**, Maxim Zdorovets, Dmitriy Shlimas; **data curation**, Malik Kaliyekperov, Natalia Volodina, Dmitriy Shlimas; **writing—original draft preparation**, Natalia Volodina; **writing—review and editing**, Dmitriy Shlimas, Rafael Shakirzyanov, Maxim Zdorovets; **visualization**, Yuriy Garanin; **supervision**, Artem Kozlovskiy and Maxim Zdorovets; **project administration**, Dmitriy Shlimas; **funding acquisition**, Dmitriy Shlimas, Maxim Zdorovets. All authors have read and agreed to the published version of the manuscript.

Conflict of Interest

The authors declare no conflicts of interest.

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