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Plastic flows and strain-induced alpha to omega phase transformation in zirconium during compression in a diamond anvil cell: Finite element simulations

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ABSTRACT

Coupled plastic flows and the strain-induced $\alpha \rightarrow \omega$ phase transformation (PT) in a zirconium sample under compression in a diamond anvil cell are investigated using finite element method (FEM). The PT is treated as strain-induced rather than pressure-induced and the previously developed model for strain-induced PTs is utilized. Very heterogeneous fields of stress tensor, accumulated plastic strain, and concentration of the ω phase are obtained for different applied loads. The PT starts at the center of a sample when pressure exceeds the minimum pressure $p_{\varepsilon}^{d} = 1.7\ GPa$, below which a direct strain-induced PT to a high pressure phase cannot occur, and it propagates from the center to the periphery with an increasing load. Even at the maximum pressure of 7 GPa, the PT is not completed everywhere. With an increasing load, the pressure and pressure gradient along the radial direction significantly increase in the two-phase region due to the much larger yield strength of the ω phase. This in turn promotes transformation and produces a positive mechanochemical feedback. Obtained results are utilized for the interpretation of published experimental data on pressure-, stress-, and strain-induced $\alpha \rightarrow \omega$ PTs in Zr and Titanium (Ti) and $\alpha \rightarrow \beta$ and $\omega \rightarrow \beta$ PTs in Zr under compression and high pressure torsion. This includes correcting the reported minimum pressures for these transformations by a factor of 3–6 due to the stress heterogeneity, the effect of transmitting media, the pressure hysteresis, and the reversibility of the transformation.

1. Introduction

The diamond anvil cell (DAC) is widely used in the field of high pressure research and it allows an in-situ study of phase transformations (PTs) under high pressure by using advanced diagnostics such as optical, Raman, and *x-ray* techniques [1–4]. Within a liquid medium, the sample is subjected to a hydrostatic load and PTs in the sample are pressure-induced. Without hydrostatic medium, or above the solidification pressure of transmitting medium, a sample in a DAC is subjected to nonhydrostatic stresses or stress tensor. When a large, irreversible reduction of the thickness takes place, material is subjected to large plastic deformation. Experiments in rotational Bridgman anvils [5,6] and rotational DAC show that plastic deformation leads to a significant reduction in PT pressure, by a factor of 2–5 in [7–12] and even almost 10 in [13,14]. Also, sometimes plastic straining leads to an appearance of new phases, which were not obtained without plastic straining [11,15,16]. Similar results in rotational Bridgman anvils were obtained for various chemical reactions [17,18]. Traditionally, PTs under compression in DAC are called "under uniaxial load," while in reality they occur under very heterogeneous multiaxial loading. PTs are characterized by transformation pressure. It depends on the degree of nonhydrostaticity, plastic strain, or plastic shear (in a rotational DAC). This is one of the main reasons (along with different concentration of impurities [19]) why transformation pressure varies in a broad range. For example, PT in zirconium (Zr) from the α phase to the ω phase under compression in DAC or metallic dies before high pressure torsion (HPT) is reported in the range from 1 to 7 GPa [19–24]. The main reason for such discrepancy was the lack of understanding that there is a fundamental difference between pressure-induced PT under hydrostatic conditions and PTs that occur during plastic deformation under high pressure, which are called in [7,10] strain-induced PTs. These two different types of PTs are introduced in [7,10]. While pressure-induced PTs start by the nucleation at the pre-existing defects (which serve as pressure and stress tensor concentrators), strain-induced PTs occur by the nucleation at new defects that are continuously generated during the plastic deformation [7,10]. Dislocations

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as the main type of defects are generated and densely piled up against grain boundaries or other obstacles during plastic flow, which creates strong concentrators of the stress tensor. The resulting local stresses near stress concentrators may be much higher than the applied pressure and may reach the level required for high pressure phase nucleation. A simple nanoscale analytical model of nucleation at the tip of the dislocation pile-up [7,10]followed by phase field simulation [25] has demonstrated that plastic straining can lead to a substitution of thermally activated nucleation with barrierless nucleation and reduce the PT pressure by a factor of 2–10 or possibly more, similar to experiments in Refs. [7–14]. Strain-induced PTs require a completely different *experimental characterization as well as a thermodynamic and kinetic description*.

A coarse graining of the above nanoscale theory [7,10] for high-pressure mechanochemistry results in a microscale, strain-controlled (instead of time-controlled), pressure-dependent kinetic equation (see Eq. (S8) in supplemental material) for strain-induced PTs. This kinetic equation represents the main way to characterize strain-induced PTs in experiments. The concept of PT pressure reduces its importance because strain-induced PT can start in quite a broad pressure range. This strain-controlled and pressure-dependent kinetic equation is included in a macroscale model that describes coupled plastic flow and PT with a sample compressed in DAC [26–29] or compressed and twisted in rotational DAC [30–33]. Analytical [7,10] and finite-element method (FEM) [26–29] have been developed and applied to investigating the evolutions of stresses, strains, and concentration of phases in the entire sample during large plastic flow and PTs under an increasing external loading. This is very important for the interpretation of the experiments because of the strong heterogeneity of all fields, which in most cases is neglected. In experiments, the pressure and concentration of high pressure phases along the radius of a sample at a contact surface has been measured only [34].

In our preceding papers [26–29], FEM was utilized for modeling and simulating the strain-induced PTs under compression in a DAC and interpreting the experimental phenomena. Coupled problems of mechanics and PTs with a large deformation were solved, which involves highly complex equations and FEM simulations. Generic material parameters have been used [26–29] to obtain some generic knowledge of PTs. In this paper, we will study the strain-induced PT in zirconium (Zr) from the α phase to the ω phase using specific material parameters from literature. Zr as a group VI transition metal has widespread applications in the space and aeronautic, biomedical, and nuclear industries. At ambient conditions, the α phase of Zr possesses the hexagonal closed-packed (hcp) structure. When the pressure is increased at room temperature, martensitic PT into the more open hexagonal structure of ω phase occurs in the pressure range of 1–7 GPa [19–24]. With a large plastic shear strain the $\alpha \rightarrow \omega$ PT occurs at around 2 GPa [35], which is considered here as a strain-induced PT, while with hydrostatic medium (4:1 ethanol: methanol) PT happens about 7 GPa [23], which is treated here as a pressure-induced PT. Under unconstrained compression before HPT of highly textured Zr in the direction orthogonal to the c-axes, $\alpha \rightarrow \omega$ PT starts at 1 GPa [24]. HPT under unconstrained conditions reduces transformation pressure in Zr down to 0.25 GPa [36]. The main problem is that in Refs. [24,36] and in many others pressure was determined as force divided by area, while it is strongly heterogeneous, with much higher pressure at the center than an average pressure. This point will be elaborated in our simulations below. Initiation of the $\alpha \rightarrow \omega$ transformation was observed at intermediate pressures between the above extremes [24,36]. For example, for nonhydrostatic compression in multianvil system, this PT occurs around 6 GPa [37]. Olinger and Jamieson reported 3.9 GPa as the PT start pressure and suggested that differences in PT start pressure for Zr in different laboratories are either due to shear stresses or oxygen content [22]. The strong effects of impurities and pressure exposure time on PT pressure for appearance of the ω phase for different materials are documented in [19]. It is known that the ω phase of Zr is preserved under normal pressure [20], which means that there is a considerable hysteresis between $\alpha \rightarrow \omega$ and $\omega \rightarrow \alpha$ transformations [38].

At temperature of 1135 K and ambient pressure the α phase transforms into the bcc structure (β phase). At room temperature and quasi-hydrostatic loading, the $\omega \rightarrow \beta$ PT has been observed [23] at pressure of about 30 GPa. The bcc β phase is unstable at the normal pressure and thus the reverse PT takes place upon unloading. However, a recent experiment has successfully stabilized the β phase at ambient conditions by using the HPT apparatus, in which five plunger turns were applied under a compression stress of 3 GPa [39]. Later, the β phase was obtained by unconstrained HPT and stabilized along with ω phase after a pressure release at 1 GPa [24] and even 0.25 GPa [36]. As we will discuss in Section 4, these numbers should be increased by a factor of 6 due to the stress heterogeneity.

In this paper, we will present the results of the FEM modeling and simulation of the strain-induced $\alpha \rightarrow \omega$ PT. In Section 2, the problem formulation is presented. In Section 3, the distributions of the components of the stress tenor, pressure, accumulated plastic strain, and concentration of the ω phase are presented in the volume of a sample at different stages of compression and analyzed. Also, pressure, shear friction stress, the yield strength of two-phase mixture, relative sliding, and normal displacement at the sample diamond boundary are given. Changes in sample thickness and maximum pressure are calculated vs. the applied load. In Section 4 the obtained results are used for the interpretation of published experimental data on pressure-, stress-, and strain-induced $\alpha \rightarrow \omega$, $\alpha \rightarrow \beta$, and $\omega \rightarrow \beta$ PTs in Zr and $\alpha \rightarrow \omega$ PT in Titanium (Ti) under compression and HPT. This includes correction of the reported minimum pressures for these PTs in Zr by a factor of 3–6 due to stress heterogeneity, analysis of the effect of transmitting media, texture, pressure hysteresis, and reversibility of the PTs. Section 5 contains concluding remarks.

2. Problem formulation

In this paper, the shapes of the DAC generally found in experiments (e.g. in [13,34,40]) will be used in our FEM models. Typically, at the pressures below 50 GPa, a flat bottom contact surface of anvil is used in experiments [13,34], and it will be used it in simulations (see Fig. 1). This is distinct from our previous model that had a bevel angle of 8.5° for the anvil contact surface under multi-megabar pressures [41]. The sample is initially pre-indented to a thickness of 50 μm at $r \leq 150 \ \ \mu m$. We do not use any gasket (similar to [14] and different from [35]), which intensifies the plastic flow by a fast reduction in sample thickness since the yield strength of the α phase is small. Axisymmetric problem formulation is used. Due to symmetry, a quarter of the sample and the anvil will be considered; the geometric parameters can be found in Fig. 1b for the anvil and Fig. 1c for the sample. We notice that with an actual sharp angle between lines CD and DE, penetration of the anvil elements into the sample elements may occur. To avoid the divergence in computations due to such a penetration, a smooth transition at point C is used by a 45° arc (see Fig. 1c).

The boundary conditions for a quarter of DAC in Fig. 1b are summarized as follows:

(1) The normal stress σ_n is applied at the top of anvil surface.
(2) The radial displacement u_r and shear stress τ_{rz} are zero at the axis $r = 0$(the line AC for the anvil and the line CF for the sample).
(3) At the contact surface CDE, the combined Coulomb and plastic friction model is applied, which will be introduced in detail in the section on friction model.
(4) At the symmetry plane $z = 0$ (the plane FH), the radial shear stress $\tau_{rz} = 0$, and the axial displacement $u_z = 0$.
(5) Other surfaces not mentioned above are stress-free.