In situ radial X-ray diffraction study of texture and stress during phase transformations in bcc-, fcc- and hcp-iron up to 36 GPa and 1000 K

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Abstract

Using a newly developed experimental protocol based on externally heated diamond anvil cells, in situ stress and textures studies are performed on polycrystalline body-centered cubic- (\(\alpha\)-), face-centered cubic- (\(\gamma\)-) and hexagonal close-packed- (\(\varepsilon\)-)Fe during plastic deformation up to 36 GPa and 1000 K. At 7 GPa and 973 K, \(\alpha\)-Fe displays a texture attributed to dominant slip on \(\{110\}\langle111\rangle\). Upon pressure increase up to 24 GPa at constant temperature, differential stress is shown to increase to \(0.6 \pm 0.1\) GPa while Fe is transforming into \(\gamma\)-Fe and \(\varepsilon\)-Fe. Textures observed in \(\gamma\)-Fe are fully induced by phase transformation, with little effect of plastic deformation. Textures observed in \(\varepsilon\)-Fe are the result of phase transformation from \(\gamma\)-Fe combined with plastic deformation, with dominant basal \(\{0001\}\langle2\overline{1}\overline{1}\rangle\) slip. The differential stress measured in \(\varepsilon\)-Fe is consistent with a shear strength of \(~1.9\) GPa at 17 GPa and 300 K, with a temperature dependence of \(~0.003\) GPa K\(^{-1}\) and a pressure dependence of 0.074.

Keywords: High pressure; Iron; Texture; Phase transformation; Strength

1. Introduction

Phase transformations in metals attract great interest because of their implications for understanding materials behavior, shape memory alloys and the fundamental physical processes driving phase transitions. In particular, phase transformations can affect the texture as crystallographic relationships and variant selection can occur between the parent and product phases \cite{1}. However, most experiments regarding textures and phase transformations are performed at ambient pressure; very little is known regarding phase transitions at pressure above 1 GPa. Similarly, strength is a fundamental property for constraining materials behavior \cite{2} and the rheology of deep earth minerals \cite{3}. As such, measurement of materials strength at high pressure and high temperature is key to understanding materials behavior under these conditions.

Recently, we have developed a new diamond anvil cell (DAC) technique allowing the study of stress and texture in polycrystals in situ up to pressures and temperatures of 50 GPa and 1500 K \cite{4}. Iron is a good candidate for testing the capabilities of this experimental technique due to its well-characterized high-pressure/high-temperature phase diagram (Fig. 1) and its importance to engineering, fundamental physics and deep earth geodynamics. Its plastic properties under high pressure have also been widely studied \cite{5-11}. Under ambient conditions, iron takes a body-centered cubic (bcc, \(\alpha\)) structure. Upon heating, it transforms to the face-centered cubic (fcc) structured \(\gamma\) phase.
This transformation in steel is of critical importance in engineering because there is a significant reduction in materials strength associated with the transformation. The high-pressure $\epsilon$ phase has a hexagonal close-packed (hcp) structure and is believed to be the main constituent of the earth’s inner core [12].

Based on considerations of crystal structures and microscopic observations, several orientation relationships for the bcc to fcc phase transformation have been proposed in the literature [13, 14] (Table 1), including the Bain [15], Kurdjumov–Sachs [16] and Nishiyama–Wassermann [17] relationships, but they have rarely been studied in situ. In this paper, we present a new experimental approach and show how it can be used for the direct in situ investigation of strength and transition mechanisms in Fe over a large portion of its phase diagram.

### 2. Experiment and data processing

#### 2.1. High-pressure/high-temperature deformation and in situ X-ray diffraction

The in situ radial X-ray diffraction ($r$XRD) and DAC deformation experiment was performed at beamline 16-BMD of the High-Pressure Collaboration Access Team (HP-CAT), Advanced Photon Source (APS), Argonne National Laboratory, and has been extensively described before [4]. Therefore, we will only provide a summary of the main relevant features.

The wavelength of the $15 \times 15$ $\mu$m$^2$ monochromatic X-ray beam was tuned to 0.3874 Å. The collection time for diffraction images was 15 min on a $3450 \times 3450$ pixel Mar345 online image plate. The sample to detector distance, detector tilt and pixel size ratios were calibrated using a CeO$_2$ standard (NIST 674b). The sample, 20 $\mu$m in thickness and 80 $\mu$m in diameter, was a pure commercial powder of iron (Alfa Aesar, 99.99% purity) with a starting grain size of 1–2 $\mu$m. Both were loaded in a high-temperature, resistive heating DAC designed for $r$XRD equipped with 300 $\mu$m tip diameter flat anvils and a gasket consisting of an amorphous boron and epoxy ring confined by a sheet of mica [4]. Heating of the sample is achieved by using 1 mm thick graphite heaters that surround the sample assembly and the tips of the diamonds. Deformation is achieved by increasing the pressure load through a membrane attached to the back of the DAC. Temperature is measured with two Pt–Pt13Rh (type R) thermocouples, with a thermocouple reading accuracy of $\pm 5$ K. Based on the differences between the readings on the two thermocouples, we estimate that our uncertainty about the temperature and temperature gradients does not exceed 60 K. Pressure is estimated from the diffraction of the sample itself, using a previously calibrated equation of state (EoS; see below). The overall experiment was performed over 37 h (Table S1).

#### 2.2. Data processing

Diffraction images were quantitatively analyzed for cell parameters, phase proportions, textures, grain sizes and lattice strains using the Rietveld method, as implemented
in the software package MAUD [26] according to the previously described procedure [8]. Representative refinement results are shown in Fig. 2 for γ- and ε-Fe at 31 GPa and 900 K. Diffraction images were integrated over 5° increments of the azimuth angle, resulting in 72 slices. Data were fitted using bcc, fcc and hcp structures with adjustable phase ratios. The variations in peak positions with azimuth were adjusted assuming the lattice strain equation of Singh et al. [27]. For the determination of crystallite size, Popa line broadening with an isotropic size–strain model was used [28], the instrument broadening having been calibrated with the CeO$_2$ standard.

Pressures are calibrated using X-ray diffraction of an internal standard, the EoS of the sample itself (iron) in the bcc structure [29], and later the hcp structure [30], to estimate the pressure at any given temperature during the experiment. To correct for the effect of non-hydrostatic stresses, pressures were calculated using the hydrostatic cell parameters of α- and ε-Fe as derived from the MAUD refinements. The temperature–pressure path explored during the experiment is shown in Fig. 1.

Differential stress was calculated using the model of Singh et al. [27] and the aggregate shear modulus of each phase. For α-Fe, it was measured under high pressure at ambient temperature [31] and under high temperature at ambient pressure [32]. Therefore, we used a simple model assuming a linear combination of both measurements. The shear moduli of ε- and γ-Fe are not well constrained under combined high pressure and high temperature. For ε-Fe, ambient-temperature ultrasonic measurements at 16.5 GPa [33] indicate that $K/G = 2.75$, while a combination of inelastic X-ray scattering and X-ray diffraction leads to $K/G = 2.62$ at 52 GPa [34]. We therefore assumed $K/G = 2.7$ for ε-Fe, calculating the aggregate bulk modulus at pressure and temperature using the EoS of Ref. [30]. There are still significant debates regarding the EoS of γ-Fe (e.g. [29]). Thus, we took an even more simplified approach, assuming that the shear modulus of γ-Fe was equal to that of α-Fe. The phase fractions, grain sizes and stresses deduced at each measured point are shown in Fig. 3. The times, pressures, temperatures, phase fractions, cell parameters, grain sizes and stresses are also listed in Table S1.

Textures were fitted using the E-WIMW algorithm with an orientation distribution function (ODF) resolution of 7.5° and assuming cylindrical symmetry about the compression direction. The ODF was further smoothed with a 10° Gauss filter in BEARTEX [35]. Axial compression textures can be compactly represented by an inverse pole figure (IPF) showing the relationship between crystallographic directions of crystallites to the compression direction. Fe has cubic or hexagonal crystal symmetry; thus, only portions of the IPF are needed to represent the ODF. Pole densities are given in multiples of random distribution (mrd), where mrd = 1 corresponds to a random

Fig. 2. “Unrolled” rXRD image (bottom) of γ-Fe and ε-Fe taken in situ at 31 GPa and 900 K with the fit from the Rietveld refinement (top). Diffraction lines from γ- and ε-Fe are labeled, and black arrows indicate the compression direction. The phase proportions are deduced from the intensity ratios between the two phases. The textures and stress are deduced from the variations in diffraction intensity and peak position with orientation, respectively.

Fig. 3. Pressure, temperature, phase volume fractions, grain size and differential stress in α- (grey-filled black circles), γ- (red filled diamonds) and ε-Fe (open blue squares) as a function of image number. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)
distribution and an mrd of infinity corresponds to a single-crystal texture.

Refinements for the bcc phase were based on measurements of the 110, 200 and 211 diffraction lines. Refinements for the fcc phase were based on measurements of the 111, 200 and 220 diffraction lines. Refinements for the hcp phase were based on measurements of the 1000, 1011, 1012 and 1120 diffraction lines. Textures measured in all three phases are summarized in Fig. 4.

3. Results

3.1. Phase diagram and phase proportions

Fig. 1 presents the pressure–temperature path explored during the experiment, while the phase proportions of α-, γ- and ε-Fe extracted from the MAUD refinements are shown in Fig. 3. The start of phase transformation between α-Fe and γ-Fe is induced at 7 GPa and 973 K. Pressure is then increased up to 24 GPa at ~980 K, with a gradual transformation of α-Fe into γ-Fe. α-Fe completely disappears at 14.5 GPa and, with further increasing pressure, we observe the gradual transformation of γ-Fe into ε-Fe. Between 24 and 33 GPa, the temperature is reduced to 900 K. It is then further reduced to 300 K while increasing the pressure to 36 GPa. This process results in further transformation of γ-Fe into ε-Fe, though a full transition to ε-Fe was never achieved.

3.2. Stress

Differential stresses measured in the three phases of Fe are presented in Fig. 3. First, there does not seem to be any significant difference between the stresses measured in all three phases. When reaching the transition to the γ phase at 7 GPa and 973 K, the stresses are low, on the order of 0.1 ± 0.1 GPa. When the pressure is increased to 24 GPa at 980 K, the stresses in the sample are 0.6 ± 0.1 GPa. The pressure is then increased to 33 GPa while the temperature is lowered to 900 K. During this process, axial strain is imposed on the sample and the stresses continuously increase to 1.3 ± 0.1 GPa. At the end of the experiment, temperature is reduced to 300 K at 36 GPa, resulting in an increase in the differential stress to about 3.0 ± 0.4 GPa.

3.3. Textures

Upon compression, α-Fe develops a texture characterized by maxima in the 001 (1.1 mrd) and 111 (2.1 mrd) directions, and a minimum at 011 (Fig. 4). At 7 GPa and 973 K, γ-Fe appears with a texture characterized by a broad, weak maximum ranging between 011 and 014, with minima at 001 and 111. Later, γ-Fe develops a weak texture with a maximum at 011 (1.4 mrd) and minima at 001 and 111. This is associated with a weakening of texture in α-Fe, with a maximum pole density of 1.8 mrd at 111 in image 62. At 16 GPa and 970 K, α-Fe completely disappears. This is correlated with the appearance of a small proportion of ε-Fe with a marked maximum at 0001 (2.25 mrd). Upon further transformation and compression, ε-Fe develops a strong texture, with a maximum at about 30° from 0001, a maximum pole density of 2.11 mrd and a distinct minimum at 0110.

4. Discussion

4.1. Phase diagram

Overall, there is a good agreement between our measurements and phase diagrams published previously [30,36]. Full phase transition to ε-Fe was never achieved. Indeed, it has been reported that kinetics of phase transitions involving γ-Fe are slow and difficult to study experi-
mentally [36,37]. Moreover, stress and pressure gradients are known to affect the pressure range of phase coexistence in Fe. Achieving a full pressure-induced phase transition is highly dependent on stress and involves significant hysteresis [38].

4.2. Stress

Very few stress measurements have been performed on Fe polycrystals in the current pressure and temperature range. In axial compression deformation experiments up to 10% axial strain in a D-DIA at 17 GPa, it was found that stresses in ε-Fe polycrystals could reach 1.6 GPa at 600 K and 1.8 GPa at 400 K [7]. At 300 K, rXRD measurements in the DAC found stresses in the order of 3.0 ± 0.2 GPa for pressures ranging between 15 and 30 GPa [6] and 4.5 ± 0.2 GPa at 52 GPa [10]. Those are all very consistent (Fig. 5) with our measurements and indicate that the strength of ε-Fe is ~1.9 GPa at 17 GPa and 300 K, with a temperature dependence of ~0.003 GPa K⁻¹ and a pressure dependence of 0.074.

4.3. Textures in α-Fe

Our measured textures in α-Fe agree with compression textures reported for bcc metals [39] and are attributed to dominant slip on [110] < 111 >. This texture is also consistent with previous texture measurements on α-Fe under non-hydrostatic stress in the DAC [20,40]. At the start of the experiment, control of the sample pressure was lost while starting the temperature increase. This resulted in a transition into ε-Fe for a brief moment (Figs. 1 and 3). However, this incursion into the ε-Fe stability field does not seem to have influenced our measurement and any effect should be negligible.

4.4. Textures in γ-Fe

After transformation, γ-Fe shows a maximum at 0 1 1 and minima at 0 0 1 and 1 1 1. Transformation textures for the bcc to fcc transformation in steel have been extensively studied and several orientation relationships for the bcc to fcc phase transformation have been proposed in the literature [13,14], including those of Bain, Kurdjumov–Sachs and Nishiyama–Wasserman (Table 1). The 0 1 1 maximum in the fcc texture can also be generated by slip on {111} < 1 1 0 > and is the typical compression texture in fcc metals [39]. It has also been observed in DAC deformation experiments on fcc Cu [41].

In order to test whether our observed texture arises from transformation or plastic deformation, we applied a transformation model based on the Nishiyama–Wasserman orientation relationships with no variant selection (Fig. 6), using the procedure described in Ref. [18]. The obtained textures are in very good agreement with experimental measurements at 16.4 GPa, after the full disappearance of α-Fe (Fig. 4). Moreover, the observed textures in γ-Fe do not evolve upon further compression. We therefore conclude that the measured textures in γ-Fe mostly result from the phase transformation and that γ-Fe was only weakly affected by plastic deformation in the experiment. Our measured textures are weak. Therefore, subtle differences between transformation mechanisms cannot be resolved and other orientation relationships such as Bain or Kurdjumov–Sachs would also fit our data.

4.5. Textures in ε-Fe

After transformation from γ-Fe, the texture in ε-Fe shows a strong maximum at 0001 that gradually transforms towards a girdle 30° away from 0001, with a pronounced minimum at 1 0 1 0. Unlike previous studies where transition occurred from the bcc phase [20,40], we do not observe a maximum at 2 1 1 0 after the phase transition, indicating a different transition mechanism. The observed girdle 30° away from 0001 is different from the pure 0001 maximum observed at 300 K in the DAC [20] and up to 600 K in the D-DIA [7]. On the other hand, it has been observed in samples undergoing laser heating at 1950 K [40]. Therefore it is possible that a different mechanism is active at higher temperatures, with a transition temperature ranging between 600 and 900 K.

The observed textures in ε-Fe could originate from a transformation from bcc to hcp or from fcc to hcp, or from plastic deformation. In order to differentiate those effects, we performed simulations of transition textures that would be obtained from the bcc phase according to Burgers orientation relationships [23], or from the fcc phase according to the Shoji–Nishiyama orientation relationships [1,24,25] (Table 1). The results are presented in Fig. 6. In both cases,
the transformation textures show a maximum at 210 with a secondary maximum 30° away from 0001, and look significantly different from the experimental results. We can therefore conclude that textures in ε-Fe are significantly affected by an orientation process, such as variant selection, nucleation or plastic deformation.

Investigation of variant selection requires data in which the effect of plastic deformation can be easily decorrelated, which is not possible with the present data. Similarly, nucleation under stress is a very plausible mechanism for high-temperature phase transformation such as the one between γ- and ε-Fe, but this goes beyond the goals of the present study.

Therefore, we decided to focus on the effect of plastic deformation and turn to polycrystal plasticity modeling. In those models, texture development depends on the deformation geometry as well as the relative activities of the different deformation modes such as slip or deformation twinning. We used the Los Alamos viscoplastic self-consistent code (VPSC, version 7) [42], which treats each grain as an inclusion in a homogeneous but anisotropic medium that has the average properties of the polycrystal. As deformation proceeds, crystals deform and rotate to generate preferred orientation. By applying different critical resolved shear stresses (CRSSs) to slip systems and twin modes, the model will favor one deformation mode over another. This results in different textures for different combinations of deformation modes.

As a starting texture for ε-Fe, we used 2000 grains weighted to resemble that obtained after phase transformation from γ-Fe (Fig. 6). The plastic properties of ε-Fe have been extensively studied under high pressure [5,7,9,11,20,40]. Among those, very few apply to ε-Fe at high temperature and we selected plastic models that generate textures closely resembling that of Fig. 3, with a maximum approximately 30° away from 0001 (Table 2). In order to match the intensity of the textures observed experimentally at the end of the experiment, we find that an incremental deformation path of 10% compressive strain is appropriate. The texture results are shown in Fig. 6, and the values for CRSS and activities of the deformation modes are given in Table 2.

Both models provide textures that broadly agree with the experimental results (Figs. 4 and 6). In both cases, deformation is strongly dominated by basal slip (61% and 99% of the total plastic activity for models 1 and 2, respectively). The rest of the deformation is accommodated by prismatic, pyramidal (h), and pyramidal (c + a) slips and twinning. It is important to note, however, that for both models the activity of tensile twinning is low. Twinning is apparently suppressed at high temperatures, as observed for Hf [5]. Indeed, in hcp metals with a low c/a ratio, tensile twinning produces a texture with c-axes nearly parallel to the compression direction, whereas a combination of basal, prismatic and pyramidal slip produces a texture with a maximum at about 30° away from the compression direction, as observed in the experiment.

4.6. High-pressure/high-temperature phase transformations in iron

Phase transformations in pure Fe are fundamental, as Fe is a model material for numerical calculations [21,43], static high-pressure experiments [20,30], shockwave physics [22], understanding the physical properties of industry-relevant materials such as transformation-induced plasticity-assisted steels [44], and because the earth’s core is believed to be composed mainly of Fe [12].

Our results show that transformation textures between α- and γ-Fe at ~970 K and pressures ranging between 7 and 14.5 GPa are in agreement with a displacive mechanism of the phase transformation and can be modeled...
using classical orientation relationships \( \text{(Fig. 4)} \). Transformation textures between \( \gamma \)- and \( \varepsilon \)-Fe above 14.5 GPa, on the other hand, do not agree with such a simple model. This differs from observations of \( \alpha \)- and \( \varepsilon \)-Fe at lower temperature, during both static loading at 300 K \([20]\) and dynamic loading at nanosecond time scales \([22]\), where orientation relationships in agreement with a Burgers-type mechanism were observed. From the present result, it is unclear whether this complexity in transformation textures towards \( \varepsilon \)-Fe is related to variant selection, another transformation mechanism or nucleation. This effect of starting crystallographic phase and temperature on transformation microstructures in \( \varepsilon \)-Fe will have to be investigated in the future.

Regarding the earth’s core, it has been suggested that phase transformations in Fe towards a cubic phase could occur \([45]\), which would explain the enigmatic observations of an innermost inner core \([46]\). Textures are important in inner core studies as they affect the shape of the measured seismic waves and induce an anisotropy in wave velocities that can be observed at a global scale \([47]\). In that regard, transformation textures in Fe should be included in core formation models \([48]\). Our results are very preliminary and cannot be integrated in such a model yet, but they pave the way towards a more materials science based understanding of this enigmatic portion of our planet.

### 5. Conclusions

An in situ study of stress and textures in polycrystalline bcc-, fcc- and hcp-Fe has been carried out during plastic deformation up to 36 GPa and 1000 K in a DAC. The start of the phase transformation between \( \alpha \)-Fe and \( \gamma \)-Fe is induced at 7 GPa and 973 K. Pressure is then increased up to 24 GPa at \( \sim 980 \) K, with a gradual transformation of \( \alpha \)-Fe into \( \gamma \)-Fe and \( \varepsilon \)-Fe. \( \alpha \)-Fe completely disappears at 14.5 GPa, and above this level we observe the gradual transformation of \( \gamma \)-Fe into \( \varepsilon \)-Fe. The temperature is then reduced to 900 K up to pressures of 33 GPa and to 300 K at 36 GPa, resulting in further transformation of \( \gamma \)-Fe into \( \varepsilon \)-Fe.

At 7 GPa and 973 K, \( \alpha \)-Fe displays a texture with maxima at 001 and 111 and with a marked minimum at 111, typical of compression textures in bcc metals. This texture is attributed to dominant slip on \( \{110\} < 111 > \). Upon phase transformation, \( \gamma \)-Fe develops a texture with a maximum near 011 and minima at 001 and 111. This texture is shown to be fully induced by the phase transformation, inherited from that of \( \alpha \)-Fe, with very little effect of plastic deformation. \( \varepsilon \)-Fe appears above 14.5 GPa, with a texture characterized by a broad maximum 30° from 0001 and a pronounced minimum near 1010. This texture is the result of a phase transformation from \( \gamma \)-Fe combined with plastic deformation, with dominant basal \( \{0001\} < 1120 > \) slip. Under differential stresses on the order of 0.2–0.6 GPa, it is shown that the effect of plastic deformation must be accounted for in order to understand the texture observed in \( \varepsilon \)-Fe, as the correspondence between transformation textures from \( \gamma \)-Fe or \( \alpha \)-Fe and experimental observations is poor.

The differential stress measured in \( \varepsilon \)-Fe is 0.6 ±0.1 GPa at 24 GPa and 980 K, 1.3 ±0.1 GPa at 33 GPa and 900 K and 3.0 ±0.4 GPa at 36 GPa and 300 K. These results, combined with previous measurements of stress in \( \varepsilon \)-Fe, indicate that, up to 1000 K and 55 GPa, the strength of \( \varepsilon \)-Fe is \( \sim 1.9 \) GPa at 17 GPa and 300 K, with a temperature dependence of \( -0.003 \) GPa K\(^{-1}\) and a pressure dependence of 0.074.

It is interesting to note that, with 10% axial strain, temperatures of \( \sim 900 \) K and differential stresses of 1.3 GPa, \( \varepsilon \)-Fe develops strong textures that are the result of plastic deformation. This implies that, in the conditions of the earth’s core, where stresses are probably low but strains can be significant, \( \varepsilon \)-Fe can develop strong textures that could be the source of the observed seismic anisotropy \([47]\). The paper also demonstrates the capability of in situ mapping of texture and stress over a wide range of pressures and temperatures. It could be applied to the in situ study of orientation relationships and variant selection during the wide range of phase transformations that can be triggered when both pressure and temperature are varied, with numerous applications in materials science, fundamental physics and geophysics.

The development of new methods, such as in situ three-dimensional X-ray diffraction (3D-XRD) or methods for intragranular orientation and lattice strain distribution determination in the DAC \([49,50]\), also offers great opportunities for understanding the physics of phase transitions under high pressure. The technique presented here can be used to study orientations in the polycrystal as a whole with good statistics. In parallel, 3D-XRD can be used to track precise orientation relationships between individual grains during the transformation. For instance, the transformation mechanism between \( \gamma \)- and \( \varepsilon \)-Fe is not well

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<th>Prismatic {1010} &lt; 1210 &gt;</th>
<th>Basal {0001} &lt; 1210 &gt;</th>
<th>Pyramidal {011} &lt; 1210 &gt;</th>
<th>Pyramidal {ε + α} {2112} &lt; 2113 &gt;</th>
<th>Tensile twinning {1012} &lt; 1011 &gt;</th>
<th>Compressive twinning {2112} &lt; 2113 &gt;</th>
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Table 2: CRSSs and activities (Act.) of the different deformation modes for model results shown in Fig. 5. Activities are shown at the end of the simulation (10% strain). CRSSs for model 1 are those of model A in Ref. \([40]\). CRSSs for model 2 are those of model 2 in Ref. \([7]\) at 600 K after 0.3 accumulated strain in grain.
understood and a combination of both methods would be a powerful tool for deciphering the effect of mechanism, variant selection and nucleation on the transition.

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.actamat.2013.04.068.

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