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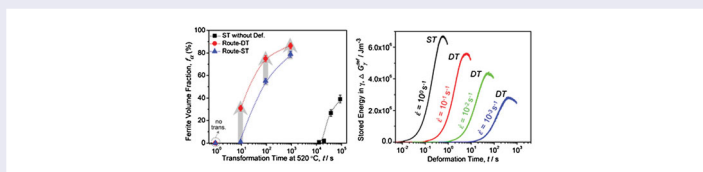
# Deformation-assisted diffusion for the enhanced kinetics of dynamic phase transformation

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## ABSTRACT

Comparison on the kinetics of two different phase transformations, including phase transformation *after* deformation and phase transformation *during* deformation (i.e. dynamic transformation, DT), reveals a new discovery that the transformation kinetics can be significantly enhanced in DT even under low driving forces. DT enables continuous generation of defects (e.g. dislocations) near the phase boundary, which can act as short-circuiting diffusion paths for atoms. The diffusivity of atoms is enhanced and the activation energy for the atom jump across the phase boundary is lowered under stress during DT, resulting in more pronounced grain growth as well as accelerated transformation kinetics.



## IMPACT STATEMENT

Deformation-enhanced grain growth is revealed in dynamic phase transformation, which will promote microstructure and property design of structural materials where phase transformations occur.

## ARTICLE HISTORY

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## KEYWORDS

Dynamic phase transformation; deformation; diffusion; kinetics; grain growth

Solid-state phase transformation commonly exists in materials and plays a major role in controlling their microstructures and properties [1–5]. Diffusional phase transformation generally incorporates nucleation and grain growth. During the nucleation, a new interface separating the product phase from the parent phase is generated. The interface migrates into the surrounding parent phase through jumps of atoms across the phase boundary (growth of nuclei) [6,7]. As in a typical diffusional phase transformation, e.g. austenite (face-centered cubic, FCC) to ferrite (body-centered cubic, BCC) transformation in steels, interstitial carbon atoms experience partitioning from ferrite to austenite by long-range diffusion. Simultaneously, individual jumps of iron and

substitutional alloying elements across the interface lead to the reconstruction of the BCC crystal from the parent FCC austenite.

The kinetics of diffusional phase transformation can be influenced by plastic deformation, which is often applied during processing of materials. The diffusivity of either interstitial or substitutional atoms could be affected by lattice defects (dislocation, vacancy, etc.) introduced by plastic deformation, since the defects may lower the planar density of atoms and provide extra free volumes that can be favorable pathways for atomic diffusion [8]. Basically, there are two different phase transformations in high-temperature processes including plastic deformation: (1) phase transformation *after* deformation of

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the parent phase (i.e. static transformation, ST) [9,10] and (2) phase transformation *during* deformation (i.e. dynamic transformation, DT) [4,5,11–15]. The deformation applied *before* or *during* transformation determines how and where the defects are introduced, and therefore could lead to different scenarios of transformation kinetics affected by nucleation density and growth rate related to atomic diffusion.

In this work, the effect of plastic deformation on the kinetics of ferritic transformation in a low-carbon steel was compared between the route-ST (i.e. phase transformation *after* deformation) and the route-DT (i.e. phase transformation *during* deformation). A surprising discovery was found: the diffusivity of atoms as well as the transformation kinetics was significantly enhanced even under low driving forces in the route-DT. This study gives a new perspective to tailor the microstructures in a wide range of materials where phase transformations occur.

The material used in this study is an Fe–10Ni–0.1C alloy (C: 0.111, Ni: 10.08, Mn: 0.01, P: 0.001, Si: 0.006, Al: 0.33, S: 0.0017, Fe: bal. (wt.%)). Para-equilibrium temperature ( $A_{p3}$ ) of the Fe–10Ni–0.1C alloy is 583°C calculated by Thermo-Calc software (time–temperature–transformation diagram is shown in supplementary Figure S1). Cylindrical specimens with a height of 12 mm and a diameter of 8 mm were machined from a homogenized plate and used for simulating the thermomechanical controlled process (TMCP) on Thermecmastor-Z (Fuji Electronic Industrial Co. Ltd.). All the specimens were austenitized at 1000°C for 300 s. In the route-ST, the austenitized specimens were cooled to 520°C at a rate of 30°C s<sup>−1</sup>, held for 60 s at 520°C to homogenize the temperature in the specimens, uniaxially compressed to a strain of 0.916 at a strain rate of 10° s<sup>−1</sup> and then isothermally held at 520°C for different periods of time followed by water quenching. In the route-DT, the austenitized specimens were cooled to 520°C at a rate of 30°C s<sup>−1</sup>, held for 60 s at 520°C, and then uniaxially compressed to a strain of 0.916 at different strain rates from 10° s<sup>−1</sup> to 10<sup>−3</sup> s<sup>−1</sup> followed by water quenching. Microstructures at the sections parallel to the compression axis were characterized by optical microscopy (OM), and a field-emission type scanning electron microscope (FE-SEM, FEI XL30S FEG) equipped with the electron back-scattering diffraction (EBSD) system. The point-counting method was used for measuring the volume fractions of ferrite on OM images. The area measured to get the ferrite fraction in the present study was 33,232.1 (μm)<sup>2</sup> for each condition.

The comparison on transformation kinetics in different processing routes is summarized in Figure 1(a), and corresponding microstructures are shown in Figure 1(b–e). More relevant microstructures can be seen in

supplementary Figures S2, S3 and S4. Here, the transformation time incorporates deformation time and isothermal holding time (after deformation). Compared to the sluggish kinetics of static ferrite transformation without deformation (indicated as ‘ST without Def.’), the kinetics of ferrite transformation in the route-ST is significantly accelerated by the deformation of austenite which could introduce a high density of defects as the nucleation sites for the subsequent ferrite transformation [9,10]. The volume fraction of ferrite in the route-ST is 0.5% at 9 s (i.e. 1 s of deformation time and 8 s of holding time after the deformation, Figure 1(b)), and reaches 79% at 916 s (Figure 1(c)). In the route-DT, the volume fraction of ferrite is around 31% at a transformation time of 9 s (corresponding to the time required for the compression deformation to a true strain of 0.916 at a strain rate of 10<sup>−1</sup> s<sup>−1</sup>, Figure 1(d)), and the fraction of ferrite dramatically increases to 86.2% at 916 s (the time for deformation at a strain rate of 10<sup>−3</sup> s<sup>−1</sup>, Figure 1(e)). It is of great interest that within the same transformation time, the volume fraction of ferrite in the route-DT is much higher than that in the route-ST, suggesting that the kinetics is significantly accelerated when the transformation occurs *during* deformation compared to that occurring *after* deformation.

The kinetics can be principally affected by the driving force for phase transformation  $|\Delta G_{\alpha\gamma}|$ , which is given by

$$|\Delta G_{\alpha\gamma}| = |\Delta G_{\alpha\gamma}^{chem}| + |\Delta G_{\gamma}^{def}| - |\Delta G_{\alpha\gamma}^{def}(f_{\alpha}) + \Delta G_{\alpha\gamma}^{int}(f_{\alpha})| \quad (1)$$

where  $\Delta G_{\alpha\gamma}^{chem}$  ( $< 0$ ) is the chemical Gibbs energy difference between ferrite ( $\alpha$ ) and austenite ( $\gamma$ ) at the same temperature,  $\Delta G_{\gamma}^{def}$  ( $< 0$ ) is the stored energy in deformed  $\gamma$ ,  $\Delta G_{\alpha\gamma}^{def}(f_{\alpha})$  ( $> 0$ ) is the summation of elastic and plastic misfit energies between  $\alpha$  and  $\gamma$  at the interface, and  $\Delta G_{\alpha\gamma}^{int}(f_{\alpha})$  ( $> 0$ ) is the free energy of  $\alpha/\gamma$  interface.  $\Delta G_{\alpha\gamma}^{def}(f_{\alpha}) + \Delta G_{\alpha\gamma}^{int}(f_{\alpha})$  includes the area density of interfaces per volume and is a function of ferrite fraction. The stored energy in austenite  $\Delta G_{\gamma}^{def}$  can be calculated by the following formula [16], assuming that the stored energy is mostly due to dislocations accumulated in the crystal during the plastic deformation,

$$\Delta G_{\gamma}^{def} = \mu b^2 \rho \quad (2)$$

where  $\mu$  is the shear modulus,  $\rho$  is the dislocation density, and  $b$  is the magnitude of the Burgers vector. The relation between flow stress  $\sigma$  of deformed austenite and dislocation density follows the Bailey–Hirsch

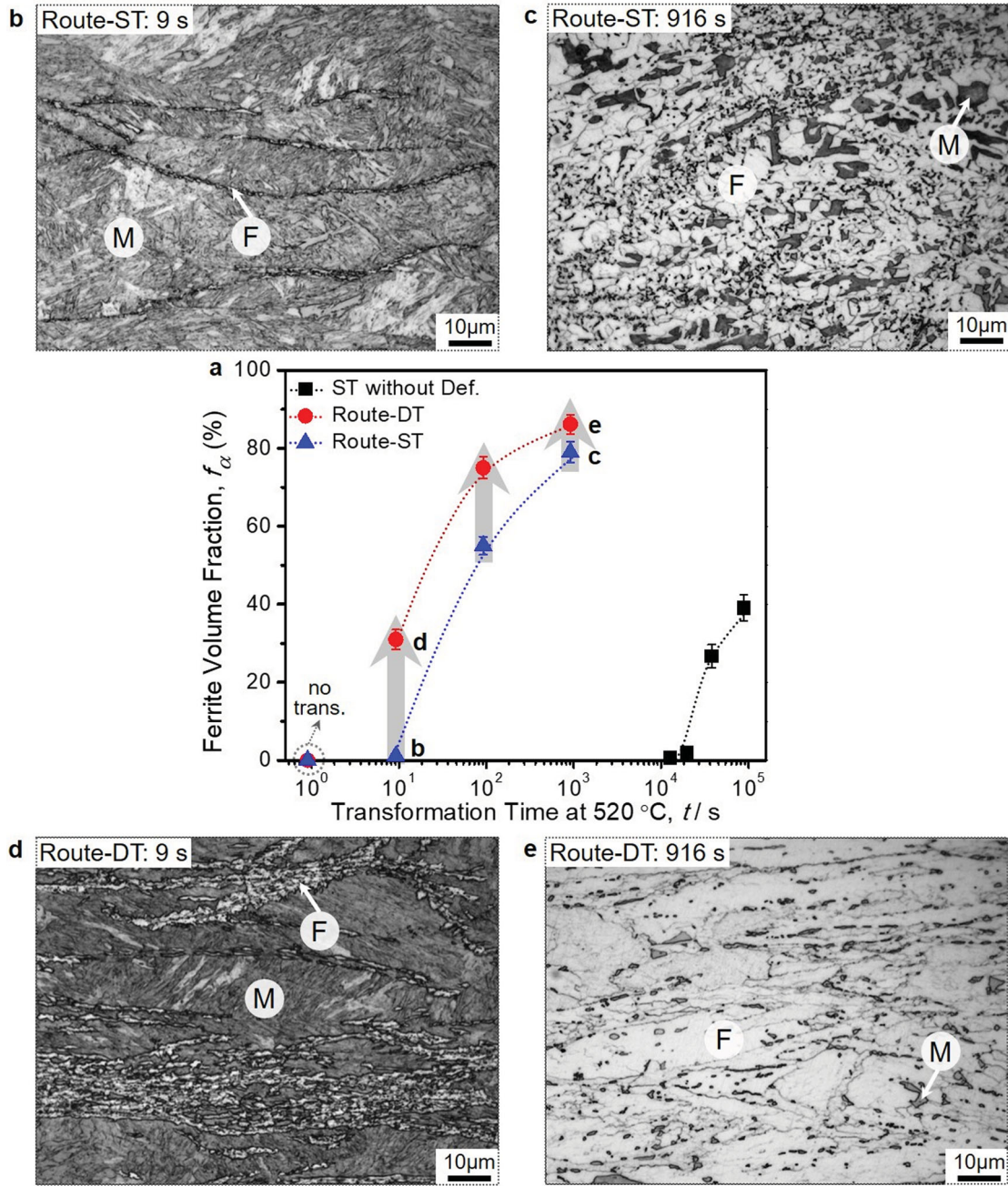


**Figure 1.** (a) Variation in volume fraction of ferrite with transformation time at 520°C. (b–e) OM images of the corresponding microstructures obtained in the two routes (ST and DT). ‘F’ and ‘M’ indicate ferrite (light area) and martensite (dark area), respectively. Compression axis is parallel to the vertical direction in the images.

equation [17],

$$\sigma = M\alpha\mu b\rho^{1/2} \quad (3)$$

where  $M$  is the Taylor factor of polycrystalline austenite, and  $\alpha$  is a numerical constant. Thus, combining Equations (2) and (3), the stored energy in austenite could be described as

$$\Delta G_\gamma^{def} = \sigma^2 / \mu M^2 \alpha^2 \quad (4)$$

In Equation (4),  $\mu M^2 \alpha^2$  is a constant, so that  $\Delta G_\gamma^{def}$  is proportional to the square of the flow stress  $\sigma^2$ . For the steel used in the present study, the values of  $M$ ,  $\mu$  and  $\alpha$  are 3.08,  $6.1 \times 10^{10} \text{ J m}^{-3}$  and 0.2, respectively [18].

Figure 2(a) shows the true stress-true strain curves of the specimens deformed at 520°C. The stored energies in  $\gamma$  calculated from Equation (4) are plotted against the deformation time in Figure 2(b). It is clear that the  $\Delta G_\gamma^{def}$  is lower in the route-DT than that in the route-ST. It should be noted that in the route-DT, austenite

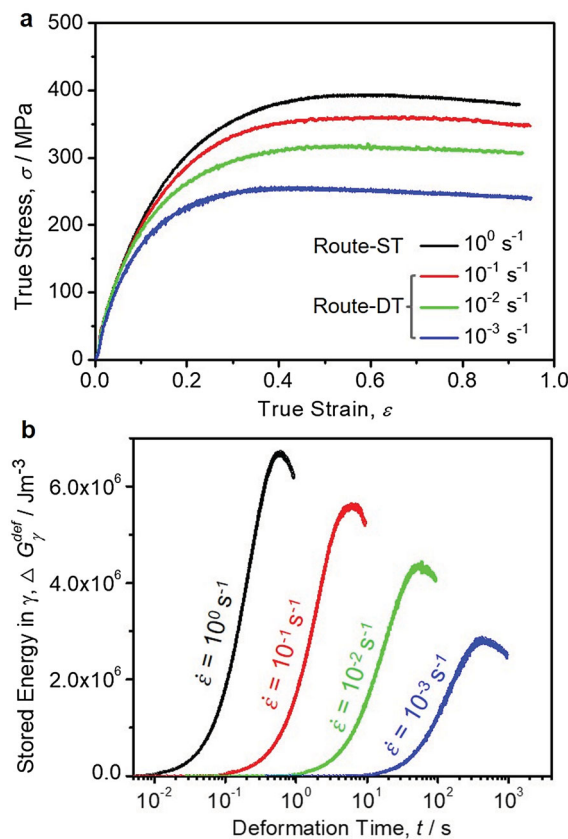


**Figure 2.** (a) True stress-true strain curves of specimens deformed to a strain of 0.916 in the route-ST (at a strain rate of  $10^0 \text{ s}^{-1}$ ) and the route-DT (at strain rates from  $10^{-1}$  to  $10^{-3} \text{ s}^{-1}$ ) at  $520^\circ\text{C}$ . (b) Stored energy in austenite ( $\gamma$ ) in the two routes, calculated by Equation (4).

to ferrite transformation occurred during the deformation. The transformed ferrite was further after its nucleation, which means that less plastic strain than the total strain was applied to  $\gamma$ . The actual stored energy in deformed  $\gamma$  is even less than that calculated by Equation (4). Results of the comparison on all the parameters in Equation (1) are summarized as follows: the chemical driving force  $\Delta G_{\alpha\gamma}^{\text{chem}}$  is equal in both routes at  $520^\circ\text{C}$ , since it only depends on the temperature; the  $\Delta G_\gamma^{\text{def}}$  is lower in the route-DT, as derived from Figure 2(b); the term  $[\Delta G_{\alpha\gamma}^{\text{def}}(f_\alpha) + \Delta G_{\alpha\gamma}^{\text{int}}(f_\alpha)]$  increases with increasing the ferrite fraction [19], so it is higher in the route-DT than in the route-ST according to Figure 1(a). Therefore, the total driving force  $\Delta G_{\alpha\gamma}$  should be smaller in the route-DT. This is a rather surprising conclusion that the transformation kinetics in the route-DT is faster than that in the route-ST even under lower driving forces.

Figure 3(a,b) shows grain average misorientation (GAM) maps of microstructures obtained at a transformation time of 916 s in (a) the route-ST and (b) the route-DT. The GAM map obtained by EBSD measurement can

be used to evaluate the degree of misorientation inside each grain [20–22]. A higher GAM value (dark red color) indicates higher misorientation within the grain. The ferrite statically transformed after plastic deformation in the route-ST incorporates mostly equiaxed grains surrounded by high-angle boundaries (HABs). The ferrite dynamically transformed in the route-DT incorporates mostly elongated coarse grains containing a large amount of low-angle boundaries (LABs). The average GAM values of ferrite transformed in the route-ST and route-DT are  $0.34^\circ$  and  $0.63^\circ$ , respectively (Figure 3(c)), indicating a more deformed ferrite structure in the route-DT. Figure 3(d) shows variations of the apparent nucleation density of ferrite in the two routes. The apparent nucleation density of ferrite was calculated by dividing the number of ferrite grains by the total area of ferrite [23,24]. As shown in Figure 3(d), the apparent nucleation density decreases with increasing the transformation time in both routes due to the growth of nucleated ferrite. Within the same transformation time at  $520^\circ\text{C}$ , the apparent nucleation density in the route-DT is much lower than that in the route-ST. This is reasonable since the higher driving force (due to higher dislocation density) in the route-ST enhanced the nucleation of ferrite. According to Figure 1(a), the ferrite fraction in the route-DT is always higher than that in the route-ST. Therefore, it can be concluded that the faster transformation kinetics in the route-DT is mainly due to the enhanced growth of ferrite during deformation.

The difference in ferrite grain growth behavior between the two routes is further discussed here. In the present study, all the experiments were conducted at  $520^\circ\text{C}$ , which is below the para-equilibrium temperature ( $583^\circ\text{C}$ ) of the steel, so the substitutional element Ni cannot fully diffuse in austenite at this low temperature. Since the transformation kinetics of static transformation without deformation is slow (shown as ‘ST without Def.’ in Figure 1(a)), carbon could be partitioned between ferrite and austenite and its long-range diffusion is considered to dominate the growth of ferrite. It is worth noting that the interaction between Ni and carbon or moving interfaces is negligible due to the low binding energies between them [25,26]. Thus, possible segregation of Ni in the interface and the solute drag effect, if any, is expected to be small in the present study.

The diffusion of carbon in austenite and ferrite can be affected by plastic deformation in terms of two aspects: strain and stress. In the route-ST, the strain of 0.916 is totally applied on austenite at a high strain rate of  $1 \text{ s}^{-1}$ . The static ferrite transformation occurs during isothermal holding after the deformation. In the route-DT at lower strain rates ( $10^{-1}$  to  $10^{-3} \text{ s}^{-1}$ ), on the other hand, the plastic deformation is firstly applied on austenite at

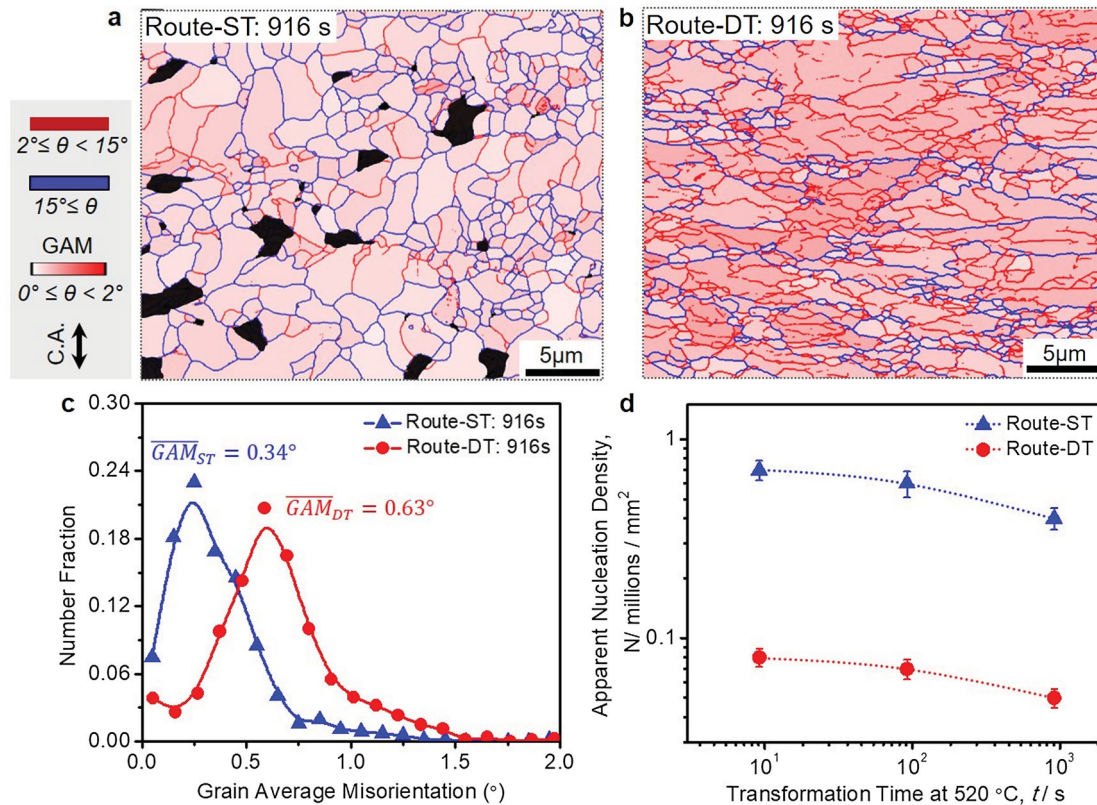


**Figure 3.** (a) and (b) are GAM maps obtained by EBSD measurements of specimens at a transformation time of 916 s in the route-ST and route-DT, respectively. Non-ferrite phases (i.e. martensite and/or retained austenite) are painted in black. LABs with misorientation of 2–15° and HABs with misorientation above 15° are drawn in red and blue lines, respectively. C.A. indicates the compression axis. (c) and (d) show distributions of GAM and variations in apparent nucleation density of ferrite grains transformed in two different routes (route-ST and route-DT), respectively.

early stages and then on dual phases after the formation of ferrite. It is, therefore, reasonable to consider that the density of the lattice defects (dislocations, etc.) in austenite, which provide potential nucleation sites for ferrite, is higher in the route-ST. Therefore, the nucleation density of ferrite was much higher in the route-ST than in the route-DT as shown in Figure 3(d). Figure 4 schematically illustrates microstructural evolutions in the two routes. Grain boundaries of austenite are strong obstacles for dislocation slips due to the crystallographic discontinuity. It is also expected that various slip systems are activated near grain boundaries for satisfying compatibility between neighboring grains. As a result, dislocation densities near grain boundaries of austenite are expected to be higher than that at grain interior (Figure 4(a)). In the route-ST, ferrite grains prefer to nucleate near grain boundaries of austenite (and along deformation bands, if any: Figure 4(b)), and consume dislocations accumulated nearby the grain boundaries (Figure 4(c)). The ferrite grains could experience early impingement due to the higher nucleation density and thus the growth of ferrite is suppressed near the austenite grain boundaries in the route-ST. The nucleated ferrite grains could grow into the

grain interior of austenite with less nucleation sites and impingement (Figure 4(c)). In the route-DT, on the other hand, the austenite is gradually strained at a slower strain rate, leading to the lower density of nucleation sites for ferrite at early stages of transformation (Figure 4(d)). The austenite and ferrite are both deformed at later stages, and dislocations are preferentially stored near austenite grain boundaries as well as austenite/ferrite phase boundaries (Figure 4(e)). The dislocations can act as effective pipes along which atoms can diffuse faster [27]. Therefore, the diffusion of carbon atoms can be enhanced through the fast routes, and thus the migration rate of austenite/ferrite phase boundaries is increased. This is the essential difference between DT and ST in ferrite growth, i.e. there are more dislocations continuously generated near the moving phase boundaries during DT, and the diffusion of carbon in ferrite and austenite can be enhanced due to these additional ‘express-ways’, resulting in more pronounced grain growth (Figure 4(f)).

The stress may also affect the growth of ferrite grains. For a single atom, the atom must pass through a thermally activated state with an activation energy to achieve an effective jump over the interface. It has been simulated

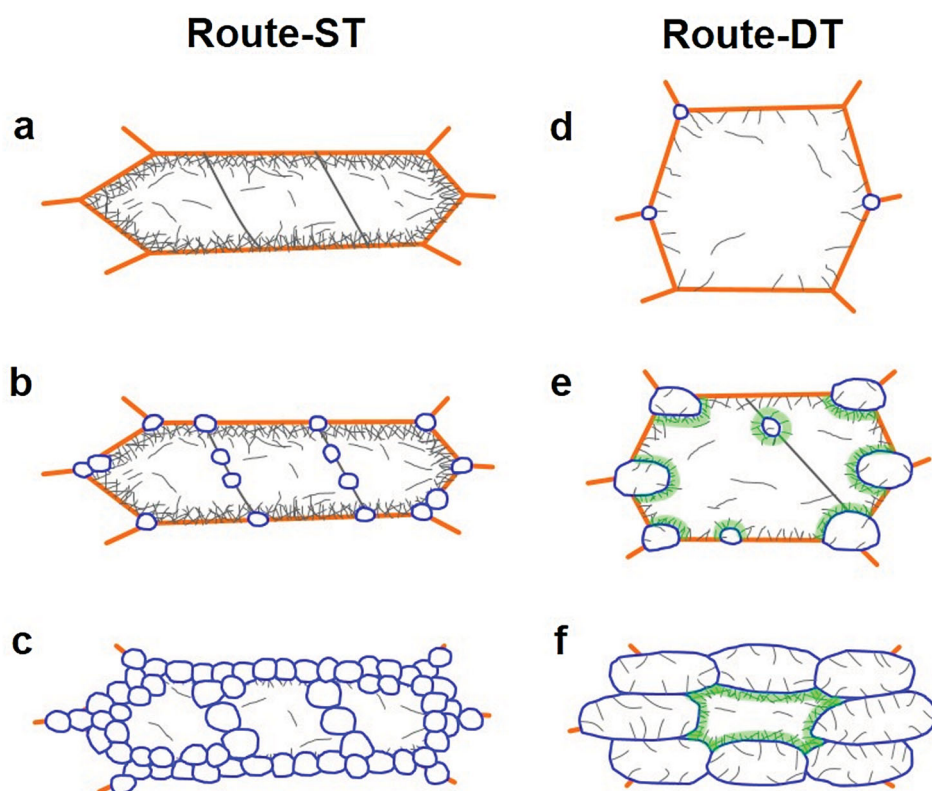


**Figure 4.** Schematic illustrations of (a–c) static ferrite transformation from deformed austenite (route-ST) and (d–f) dynamic ferrite transformation (route-DT). Orange and blue lines depict austenite and ferrite grains, respectively. Black lines represent defects (mainly dislocations). The areas where atom diffusion is enhanced are highlighted by green color in (e and f).

[8] that under a compressive stress, the presence of lattice defects (vacancy, etc.) near austenite/ferrite interfaces could reduce the planar density of atoms, providing extra free volume at moving interfaces, and thereby lowers the extent of local rearrangement necessary to move the interface. Different from the route-ST where the transformed ferrite is almost free of defects (Figures 3(a) and 4(c)), the ferrite in the route-DT is continuously deformed during transformation (Figures 3(b) and 4(f)). The introduced lattice defects could enhance the accommodation of jumping atoms over the phase boundary under stress, and then increase the migration rate of the phase boundary (i.e. growth rate of ferrite).

In summary, our findings reveal a significant enhancement of transformation kinetics in dynamic phase transformation even under low driving forces. This is attributed to the enhanced diffusivity of atoms in the migration of phase boundary (i.e. growth of ferrite) under strain and stress applied by continuous deformation. The strain-enhanced diffusivity of atoms results primarily from dislocation-assisted diffusion through short-circuiting paths (pipes) near austenite/ferrite interfaces. Stress may lower the activation energy for atoms to jump across phase boundaries. This discovery could lead to new strategies for tailoring required microstructures and

properties in many materials where phase transformation occurs.

### Disclosure statement

No potential conflict of interest was reported by the authors.

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