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Comment on "Cryoforged nanotwinned titanium with ultrahigh strength and ductility"

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We analyze the results of Zhao *et al.* (Reports, 17 September 2021, p. 1363) with a focus on the mechanical properties and microstructural evolution. We conclude that their results, together with the explanations and interpretations, are confusing, misleading, or even wrong.

The mechanical properties of the cryoforged Ti were shown in figure 2 of Zhao *et al.* (*1*). Of particular interest, also the focus of the report, was the true stress-strain curve of the "nanotwinned Ti." However, many essential parameters, such as the ultimate tensile stress (UTS), fracture strength, uniform elongation, etc., must be obtained from the tensile engineering stress-strain curve (ASTM Standard E8/E8M). The relationship between the engineering quantities and the true quantities can be expressed as $\sigma_T = \sigma_e(1 + \varepsilon_e)$ and $\varepsilon_T = \ln(1 + \varepsilon_e)$, where the subscript "T" refers to "true" and "e" to "engineering." As such, the claimed 2-GPa UTS is misleading. So that the UTS of the specimens under different loading conditions could be derived, figure 2 of (*1*) was used to plot the engineering stress-strain curve, as shown by Fig. 1 here. At 77 K, the maximum stress of the "nanotwinned Ti" is barely above 800 MPa. In other words, the actual UTS of Zhao *et al.*'s "nanotwinned Ti" is merely ~800 MPa at 77 K.

From these engineering stress-strain curves, the following can be further observed. First, the elongation to failure (EL%, another way to designate tensile ductility) of the "nanotwinned Ti" reached more than 160%. This exceedingly high apparent tensile ductility is most probably caused by the specimen size effect (*2*, *3*). The supplementary materials of Zhao *et al.* showed that the gauge length of the "nanotwinned Ti" was only 0.68 mm, confirming our interpretation of the 160% elongation to failure at 77 K. The nonstandard specimen design with such aspect ratios will surely introduce error of strain calculation according to the finite element analysis of (*2*). In the supplementary materials of (*1*), Zhao *et al.* claimed that "At cryogenic temperature, the strain was derived from the recorded displacement. Stiffness correction of the stress-strain relations from the cryogenic tensile tests were conducted using the relationship of the displacement and the digital-imaged correlated (DIC)–measured strain obtained from room-temperature tests." However, this methodology should not be taken as justified to derive the true deformation of the specimen gauge section for two reasons. First, the "relationship of the displacement and the DIC-measured strain" is believed to be closely related to the deformation behavior of the tested material. But the mechanical responses of "nanotwinned Ti" were quite different between 77 K and room temperature (RT), which could lead to significant difference in the so-called relationship. Second, the apparent uniform elongation was much larger at 77 K than that at room temperature. Calibrating the large deformation by a small portion of the curve could lead to another dimension of uncertainty.

The engineering stress-strain curve of the "nanotwinned Ti" at RT exhibited flow softening almost immediately after yielding. The complete absence of twinning at RT that showed so strong an effect at 77 K is hard to understand, as Paton and Backofen observed twinning activities in pure Ti even at 673 K in their classical work (*4*). To claim that the "cryoforged Ti" is thermally stable at 673 K and at the same time to presume that detwinning is responsible for the lack of strain hardening in the "nanotwinned" specimen at RT is not very consistent. Neither is it convincing. Alternatively, the UTS of their "nanotwinned Ti" can be derived from their own true stress-strain curves and strain–hardening rate curves. To illustrate this, their true stress-strain curves and strain–hardening rate curves could be combined. According to the Considère criterion (*5*), necking starts at UTS. The UTS of the "nanotwinned Ti" thus obtained is only ~820 MPa, at sharp contrast with the authors' claim that the UTS of their "nanotwinned Ti" was ~2 GPa at 77 K. Again the "impressive" uniform elongation was nothing but an artifact due to the tensile specimen design. It is entirely different from the results described in

the report.

To verify our comments and concerns, we built a finite element (FE) model to reproduce the tensile process of the "nanotwinned" and coarse-grained Ti (Fig. 2A). The dimension of the model specimen was from Zhao *et al.* The boundary and loading conditions were made similar to those provided in Zhao *et al.*'s paper as much as possible. The gauge section length was 0.65 mm and the overall length was 1.78 mm. The overall deformation was that of the overall length, which was used as the overall displacement of the test machine or the cross-head displacement. It should be noted that there were other contributions to the apparent displacement in the real test, indicating that even larger error in strain measurement might be involved in the real test than in the FE simulations presented here. The material's constitutive parameters used in the FE model were derived directly from the true stress-strain curves of Zhao *et al.* Mesh independence was verified before the simulations. Figure 2, B to D, presents the distribution of equivalent plastic strain in the tensile specimen at 77 K at different strains. Nonuniform deformation was obvious, which deviated from the uniaxial stress state that the tensile tests should comply with. The stress triaxiality η, defined as the hydrostatic pressure divided by the Mises stress, along the center line of the specimen is given in Fig. 2E. The value of η was ~0.4 to 0.5 within the gauge section and was much larger than that for uniaxial tension (η = 0.33). These simulation results clearly demonstrate that the tensile testing in Zhao *et al.* could not provide the generally accepted evaluation of strength and ductility, and comparison of their strength and ductility results with those from the standardized experiments would be meaningless. The strain of the specimen could also be reexamined by FE analysis. By our calculation, the deformation of gauge section Δl_1 was almost the same as that of the whole specimen Δl_2 at RT (with $\Delta l_1 \approx 0.92\Delta l_2$). However, this relation becomes $\Delta l_1 \approx 0.62\Delta l_2$ at 77 K. On the basis of the simulations, this could have resulted in ~50% overestimation of the deformation, which in turn will produce 50% overestimation of the engineering strain and 23% overestimation of the true stress.

The next observation we found troubling is related to figure 3 of Zhao *et al.*, particularly the electron backscattered diffraction (EBSD) images of the "nanotwinned Ti" specimen at 77 K. These images (A to L), according to the authors, were the microstructure evolution of the "nanotwinned Ti" specimen under cryotension. Our strong concerns with regard to these images are given below, focusing on images A to F, although they equally apply to images G to L. First, these images, according to Zhao *et al.*, were taken at different engineering strains of the specimen, from 0% to 35%. However, it is fair to say from these images that the only change was the density of twins with increase in strain. The shape, orientation, and dimension of the grains were nearly the same at all strains up to 35%, the maximum presented by the authors. In other words, the microscopic strains at all the "macroscopic" strains were vanishingly small. To illustrate this more clearly, we traced out the central yellow grain in these images and compiled the tracings in one figure (Fig. 3). Obviously, nothing seemed to have happened to this grain during tension to 35% engineering strain. How can the authors reconcile the nearly zero microscopic strains with the increasingly large macroscopic strains? Second, the gauge length of their cryotension specimen was only 680 μm, and the yellow grain was at least 150 μm in size, more than 20% of the total gauge length. The height of the individual images was ~300 μm, which did not show plastic deformation at all except for the increasing "twin activities." In other words, if this region showed nearly zero strain, what exactly did the twins and dislocations do to result in 35% macroscopic strain? Third, according to the authors, there were both "$\{11\bar{2}2\}$ compression twins with a misorientation of ~65° (green), and $\{10\bar{1}2\}$ tensile twins with a misorientation of 85° (orange-yellow)" (excerpted from the caption of figure 3 of Zhao *et al.*). However, during tensile loading, the boundaries of these preexisting twins did not change at all. According to Wang *et al.* (*6*), the critical resolved shear stress (CRSS) of $\{10\bar{1}2\}$ twins in coarse-grained pure Ti is only ~200 MPa. The stress level of the "nanotwinned Ti" at 77 K is at least three times the CRSS. At such a high stress level, how could these twins remain stationary? Regarding the $\{11\bar{2}2\}$ contraction twins, Guo *et al.* (*7*) used in situ micropillar compression of pure Ti to show that an applied stress as low as 100 MPa resulted in deformation twin nucleation. Their experiments were similar to the classical in situ experiments performed by Price on cadmium and zinc (*8–11*) in that the initial crystal for deformation was nearly defect-free and the nucleation of twins was homogeneous, which needed a much higher stress than heterogeneous twin nucleation (*12*). It is known that the growth of deformation twins requires lower stresses than twin nucleation. In other words, the stress needed for the $\{11\bar{2}2\}$ contraction twins to grow in pure Ti should be lower than 100 MPa. Thus, it is confusing to observe that nothing had happened to the $\{11\bar{2}2\}$ twins during the tensile straining of the specimen in figure 3 of Zhao *et al.*

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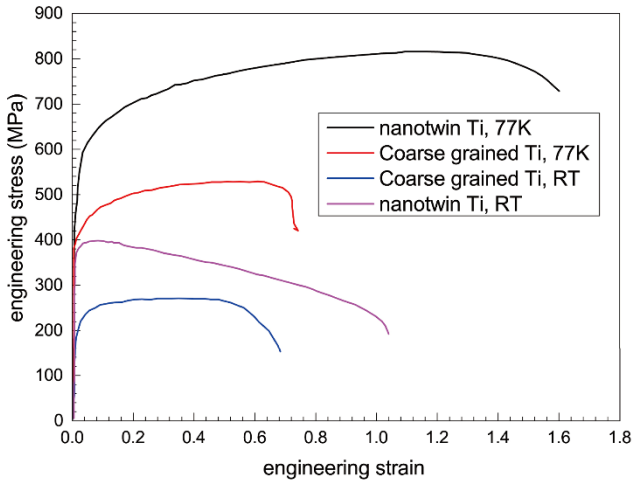

Fig. 1. Engineering stress-strain curves derived based on the true stress-strain curves of Zhao *et al.*

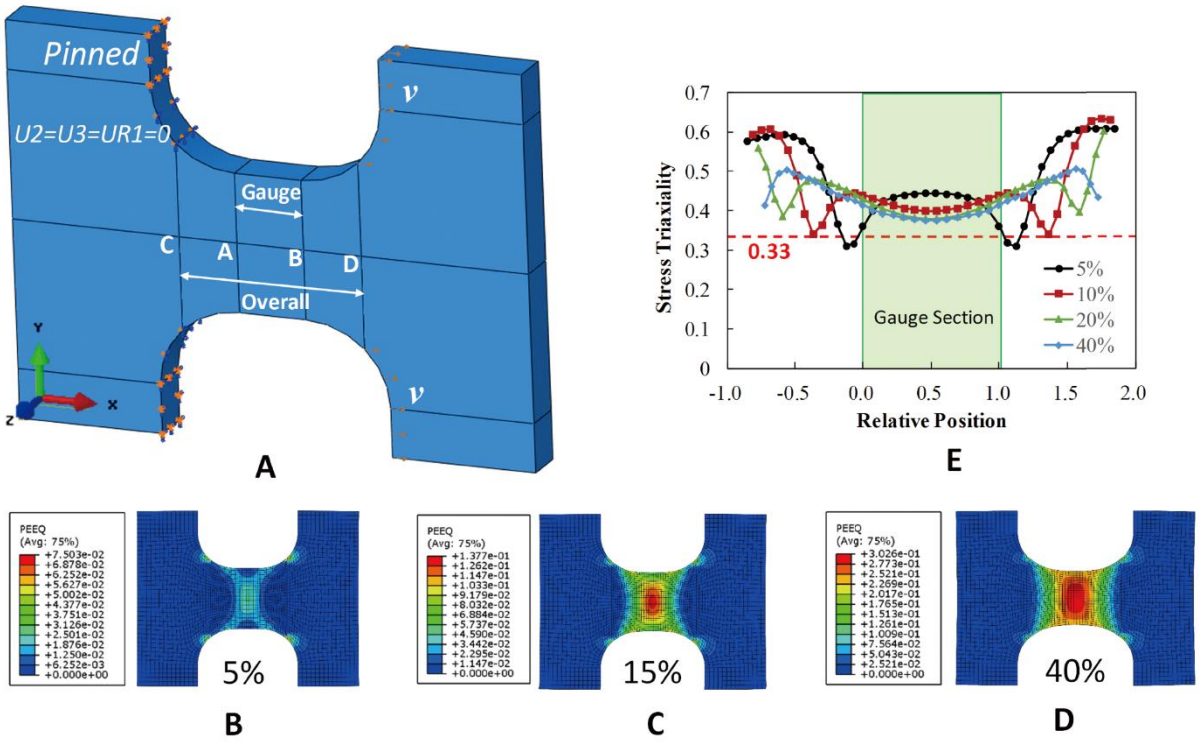

Fig. 2. Finite element (FE) simulation of tensile deformation. (**A**) FE model of the tensile specimen. The dimensions of the specimen are from Zhao *et al*. The boundary condition and load simulate those from the tests. The deformation of overall length *CD* is used to represent the measured crosshead displacement. (**B** to **D**) FE simulation results: Distribution of equivalent plastic strain over the short specimen under tension at 77 K. The percentage indicates the applied engineering strain. Nonuniform deformation is obvious. (**E**) FE simulation results: the stress triaxiality distribution along the axis of a short specimen. The percentage indicates the applied engineering strain. Deviation from the uniaxial tensile state (where stress triaxiality is 0.33) is significant.

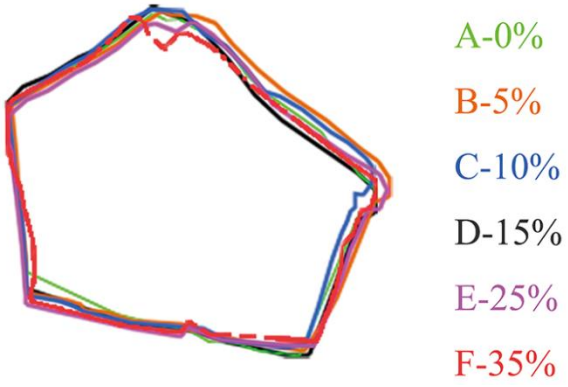

Fig. 3. The traced-out grain in the center of images A to L in figure 3 (yellow in images A to F) of Zhao *et al*. The corresponding alleged strains were also given along with the grain.

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