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## High Strength and High Uniform Ductility in a Severely Deformed Iron Alloy by Lattice Softening and Multimodal-structure Formation

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Despite high strength of nanostructured alloys, they usually exhibit poor uniform ductility. For many applications, it is an important issue to design new nanostructured alloys which have both high strength and high uniform ductility. In this study, an Fe–Ni–Al–C alloy with ultrahigh tensile strength of 1.9–2.2 GPa and high uniform ductility of 16–19% was developed by concurrent employment of several strategies: (i) appropriate choice of chemical compositions for lattice softening, (ii) severe plastic deformation using the high-pressure torsion method for grain refinement, and (iii) control of strain level for multimodal-structure formation composed of equiaxed nanograins, lamellar coarse grains and fine precipitates.

**Keywords:** Ductile Nanostructured Steels, Ultrafine-grained (UFG) Materials, Severe Plastic Deformation (SPD), Martensitic Phase Transformation, High-pressure Torsion (HPT)

Bulk nanostructured metallic materials have very high strength, but they exhibit poor strain hardening and low uniform ductility because of limitations in the accumulation of dislocations. Several strategies such as severe plastic deformation (SPD), [1–4] nanotwinning, [5–8] lattice softening by composition control, [9–13] bimodal microstructure formation, [14–16] introduction of lamellar microstructure, [17] introduction of precipitates, [18–20] transformation-induced plasticity (TRIP) [21,22] and twinning-induced plasticity (TWIP) [23–25] have been employed for improvement of ductility of nanostructured materials. However, despite these attempts, there has been little progress in development of nanostructured materials with ultrahigh strength and high uniform ductility. In this study, lattice softening is concurrently employed with several other strategies and an Fe–Ni–Al–C alloy with ultrahigh tensile strength of 1.9–2.2 GPa and high uniform ductility of 16–19% is developed.

In the Fe–Ni system, when the chemical composition is set to some critical levels as Fe–36.5%Ni (in mol%), the material exhibits lattice softening and the value of elastic constant ( $C_{11}$ – $C_{12}$ ) and shear modulus  $G$  in the  $\langle 110 \rangle$  direction reach minimum levels as 43 and 29 GPa, respectively. [26] These levels are significantly smaller than ( $C_{11}$ – $C_{12}$ ) = 95 GPa and  $G_{\langle 110 \rangle \{111\}}$  = 70 GPa for pure Fe. [26] At the critical composition for lattice softening, the stability of the  $\gamma$  phase with the fcc structure decreases and the martensite phase ( $\alpha'$  phase) becomes stable at temperatures close to room temperature. [26] The lattice-softened alloys usually exhibit high strength and good plasticity because of elastic anisotropy and phase instabilities. [9–13]

One approach to design new lattice-softened alloys with ultrahigh strength is the addition of extra alloying elements and examining the stability of phases. In an attempt to increase the strength of the Fe–Ni system, Co as a stabilizer of the  $\gamma$  phase and Ti as a stabilizer of the

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bcc phase were added to the Fe–Ni system.[10,13] The critical composition for lattice softening was determined to be Fe–18%Ni–36%Co–8%Ti (in mol%). Although the material exhibited ultrahigh tensile strength of 3 GPa and high elongation to failure of 10%, its uniform ductility was negligible.[13] It appears that a combination of lattice softening with other strategies is essential to design new alloys with ultrahigh tensile strength and high uniform ductility.

In this study, C as a stabilizer of the  $\gamma$  phase and Al as a stabilizer of the bcc phase are added to the Fe–Ni system and the material is subjected to SPD to form a multimodal structure composed of equiaxed nanograins, lamellar coarse grains and fine precipitates. Al and C were deliberately selected to form a Ni–Al–C second phase. The formation of this second phase is important to form fine precipitates, to form lamellar structure during deformation and to avoid formation of uniform nanograins by SPD processing.[27] The best composition that exhibits both lattice softening and formation of the second phase is Fe–24.6%Ni–5.8%Al–0.44%C (in wt%).[28] For this composition, the martensite phase becomes stable at room temperature (see a large fraction of the  $\gamma$  phase and a small fraction of the  $\alpha'$  phase after solution treatment in Figure 1) and an appreciable amount of second phase are formed (see Al–Ni-rich phase in Figure 2(a)).

For experiments, the Fe–Ni–Al–C alloy, with  $\sim 40$   $\mu\text{m}$  grain size, was prepared by melting under Ar atmosphere, subsequent forging at 1423 K, and solution treatment at 1373 K for 86.4 ks. In order to increase the strength, SPD using a method called high-pressure torsion (HPT) [29–33] was introduced on disc specimens (10 mm diameter, 0.8 mm thickness) by compression under a pressure of 6 GPa and concurrent rotation of 0.5, 1, 10, and 50 turns. The samples were evaluated by means of Vickers microhardness measurement, X-ray diffraction (XRD) analysis using the Cu K- $\alpha$  radiation, tensile testing (1.5 mm gauge length,

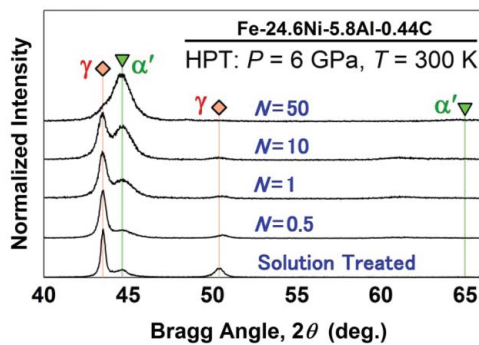


Figure 1. Phase transformation from  $\gamma$  phase to  $\alpha'$  phase during HPT processing. XRD profiles for samples processed by HPT for 0.5, 1, 10 and 50 turns including solution-treated sample.

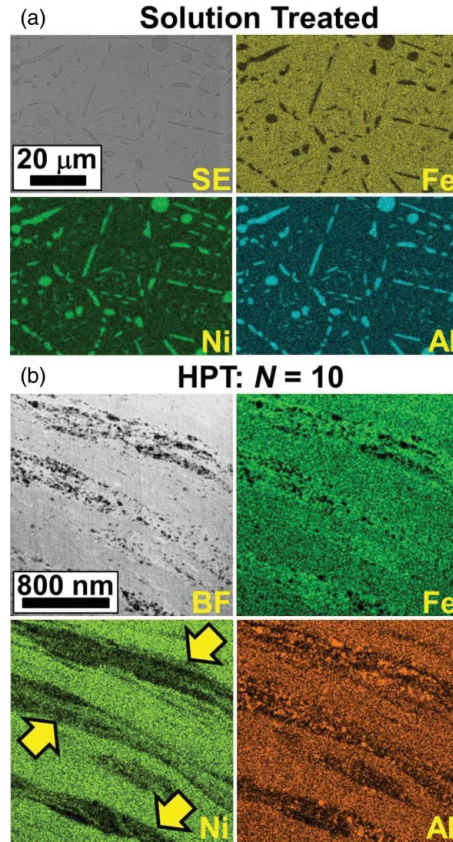


Figure 2. Distribution of elements Fe, Ni and Al before and after processing by HPT. (a) SEM-EDS mappings for solution-treated sample, (b) STEM-EDS mappings for sample processed by HPT for 10 turns.

0.7 mm gauge width, and 0.7 mm thickness), scanning electron microscopy (SEM), transmission electron microscopy (TEM), scanning transmission electron microscopy (STEM), selected-area electron diffraction (SAED) analysis, and energy-dispersive X-ray spectrometry (EDS). For TEM and STEM, thin foils were prepared from the discs at 3 mm away from the disc center with a focused ion beam (FIB) system. The uniform ductility in this study was directly measured from the tensile curves, however since the size of the tensile specimens was small, the effect of elastic deformation of grips was cancelled out using the following relationship:

$$\Delta l_s = \Delta l_T - \Delta l_M = \Delta l_T - qF. \quad (1)$$

In this equation,  $\Delta l_s$  is the real elongation of the specimen,  $\Delta l_T$  is the total measured elongation,  $\Delta l_M$  is the elongation arising from the machine and grips,  $F$  is the load and  $q$  is a constant depending on the geometry and stiffness of the machine and grips. In this study,  $q$  was determined by comparing the slope of the tensile curves in the elastic region with the elastic modulus of the material (175 GPa).

Following the HPT processing, a  $\gamma \rightarrow \alpha'$  phase transformation occurred, while the fraction of  $\alpha'$

phase increased with an increase in the shear strain (i.e. increasing the number of HPT turns), as shown in Figure 1. The homogeneous formation of equiaxed nanograins (steady-state condition), which occurred only after a few HPT turns in the Fe–Ni–Co–Ti alloy,[13] occurred only after very large shear strains (after 50 turns) in the Fe–Ni–Al–C alloy because of the presence of second phase (the steady state is achieved after a few turns in most single-phase metallic materials [32,33]). The microstructure of the sample after HPT processing for 10 turns consists of fine Al-rich precipitates with sizes in the range of 2–90 nm as in Figure 2(b), equiaxed nanograins with 10–20 nm grain size as in Figure 3(a) and coarse-grained lamellar structure with up to 1  $\mu$ m grain length as in Figure 3(b). The width of lamellar structures ranges from 10 to 50 nm, but the lamellae exhibit low angles of misorientation in most regions as in Figure 3(b). The formation of nanograins and lamellar structure in the tensile direction can be seen more clearly from Figure 3(c) and 3(d), respectively. It is interesting that the areas close to the precipitates, as indicated by arrows in Figure 2(b), are depleted from the Ni atoms after processing by HPT.

Some representative stress–strain curves of the Fe–Ni–Al–C samples processed by HPT for 0.5, 1, 10, and 50 turns are delineated in Figure 4(a) from tensile testing conducted at room temperature and

with an initial strain rate of  $2 \times 10^{-3} \text{ s}^{-1}$ . The tensile strength and ductility are 0.8 GPa and 40% for the coarse-grained solution-treated alloy, while the tensile strength increases and uniform ductility decreases after processing by HPT because of the formation of nanograins. The samples processed for 0.5, 1, and 10 turns exhibit excellent combinations of ultrahigh strength (1.9–2.2 GPa) and high uniform ductility (16–19%) because of having multimodal microstructures with  $\gamma + \alpha'$  crystal structures. However, the sample processed for 50 turns exhibits little ductility because of the formation of uniform nanograins of mainly  $\alpha'$  phase. Since the samples processed for 50 turn broke

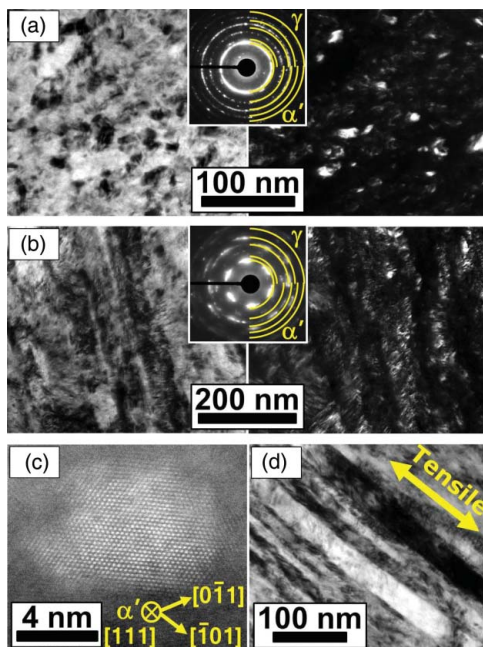


Figure 3. Heterogeneous microstructure of sample processed by HPT for 10 turns. (a, b) TEM bright-field images (left), corresponding dark-field images (right) and SAED patterns (center) taken from two different regions, (c) STEM bright-field lattice image of a nanograin with  $\alpha'$  phase, (d) TEM bright-field image of lamellar grains formed in tensile direction.

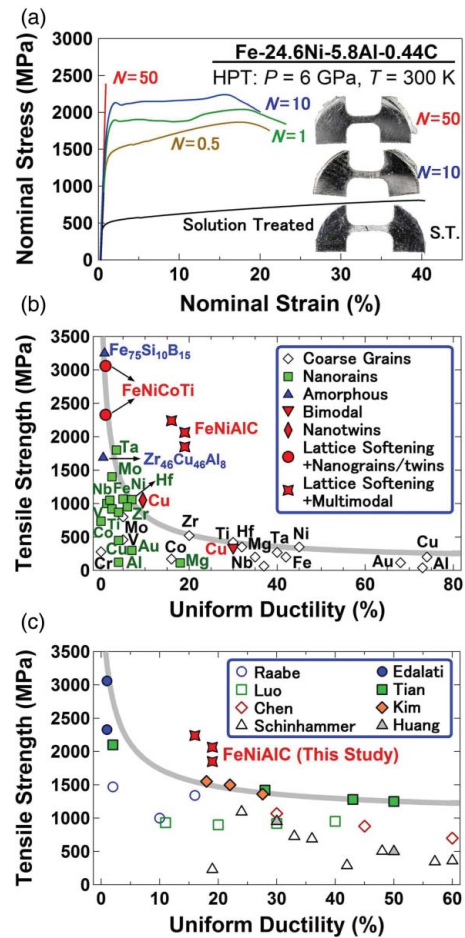


Figure 4. Superior tensile properties of Fe–Ni–Al–C sample processed by HPT. (a) Nominal tensile stress versus nominal tensile strain curves for samples processed by HPT for 0.5, 1, 10 and 50 turns including solution-treated sample, (b) plot of ultimate tensile strength versus uniform ductility for Fe–Ni–Al–C samples including data for coarse-grained pure metals, nanograined pure metals, two amorphous materials,[34,35] Cu with nanotwins,[5] Cu with bimodal microstructure [15] and Fe–Ni–Co–Ti alloys with lattice softening and nanotwins,[13] and (c) plot of ultimate tensile strength versus uniform ductility for Fe–Ni–Al–C samples including data for several steels with high strength and high ductility.[8,13,20–25]

in the elastic region during the tensile test, its tensile strength was estimated from hardness measurement as  $HV/3$  ( $HV = 730$  Hv). In order to improve the ductility of the samples processed after 50 turns, the sample was annealed at 373–673 K for 3.6 ks. The samples exhibited a significant hardening by annealing to a hardness level of 950 Hv (corresponding to a tensile strength of  $HV/3 = 3.1$  GPa) but with little ductility.

The tensile testing results shown in Figure 4(a) are summarized in Figure 4(b) in comparison with relevant data including coarse-grained pure metals produced by high-temperature annealing, ultrafine-grained pure metals produced by HPT, two amorphous materials,[34,35] a sample of Cu with bimodal microstructure,[15] a sample of Cu with nanotwins [5] and two lattice-softened Fe–Ni–Co–Ti samples with nanograins and nanotwins.[13] A solid line in Figure 4(b) represents the general relation between the strength and the ductility, indicating that there is a trade-off relation between the strength and the ductility for all data points. However, the present Fe–Ni–Al–C alloy provides excellent combinations of ultrahigh strength and high ductility when compared to any other materials. As shown in Figure 4(c), the current Fe–Ni–Al–C alloy exhibits a better combination of ultrahigh strength and high ductility even when compared to other steels with high strength and high ductility such as TWIP and TRIP steels.[8,20–25] It should be noted that care is required for comparisons in Figure 4(c), because the gage length is as small as 1.5 mm and the gage length-to-width ratio and the gauge length-to-thickness ratio are  $\sim 2$  in this study. These dimensions of the tensile specimens may result in an overestimation of the tensile ductility.[36] It should be noted that the uniform ductility in this study was directly measured from the tensile curves using Equation (1), but an examination of the length of the samples after the deformation, as shown in Figure 4(a), confirms that the measurements are reasonable.

In order to understand the deformation mechanism, tensile specimens were made from the solution-treated sample and the HPT-processed samples ( $N = 10$  and 50) and pulled to normal strains of 20% for the solution-treated sample, 10% for  $N = 10$  and to failure for  $N = 50$ . Thin foils were prepared from these specimens after tensile test using an FIB system and the microstructures were examined. For the sample before processing by HPT, many twins form during the tensile test, as shown in Figure 5(a) and 5(b), suggesting that high ductility of this sample ( $\sim 40\%$ ) should be due to the TWIP effect. For the HPT-processed sample, the formation of twins is not detected during the tensile test, but large fractions of dislocations, as shown in Figure 5(c) and 5(d), are formed in the coarse grains. Although the micrograph in Figure 5(c) corresponds to a single grain, the contrasts corresponding to dislocations accumulations can be seen clearly in the micrograph. The presence

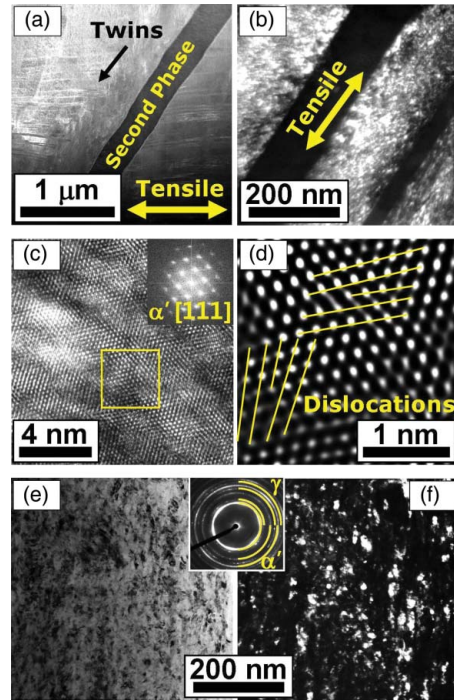


Figure 5. Microstructure of samples processed by (a, b) solution treatment and pulled in tensile test for 20%, (c, d) HPT for 10 turns and pulled in tensile test for 10% and (e, f) HPT for 50 turns and broke in elastic region in tensile test. (a) is STEM bright-field image, (b) is TEM dark-field image of twins, (c) is high resolution TEM image and corresponding diffractogram, (d) is lattice image of two edge dislocations obtained by inverse fast Fourier transform from regions marked by square in (c), (e) is bright-field image and corresponding SAED pattern, and (f) is dark-field image of nanograins.

of edge dislocation in the HPT-processed sample after the tensile testing is shown more clearly in Figure 5(d). It should be noted that the uniform variations of  $d\sigma/d\epsilon$  ( $\sigma$ : stress,  $\epsilon$ : strain) for the solution-treated sample (see Figure 4(a)) suggest that the deformation is controlled mainly by a single mechanism as twining. However, the non-uniform variations of  $d\sigma/d\epsilon$  for the HPT-processed sample suggest that different single or coupling mechanisms contribute to the formation and accumulation of dislocations and resultant combination of ultrahigh strength and high ductility. For the sample processed by HPT for  $N = 50$ , which broke in the elastic region, only nanograins present in the microstructure (see Figure 4(e) and 4(f)). The brittle fracture of this nanograined sample is due to the reduction of toughness and formation of tiny cracks. The excellent mechanical properties in this study should be due to several reasons, as outlined below: lattice softening,[9–13] grain refinement by SPD,[1–4] coexistence of nanograins and coarse grains, [14–16] and formation of lamellar structures with fine precipitates.[17,18]

The materials having softer lattice generally exhibit higher ductility. However, these particular lattice-softened materials also exhibit an unusual high

strength. Saito et al. suggested when  $G$  decreases by lattice softening, the ideal shear stress decreases and becomes comparable to the local deformation stress and this leads to a dislocation-free plastic deformation.[9] Another study, which is more consistent with the current observations, showed that spreading of the core radii of dislocations occurs by lattice softening and this leads to lower mobility of the dislocations.[12] Li et al. also suggested that the movement of dislocations can be pinned with a low density of lattice obstacles in lattice-softened materials because of significant elastic anisotropy and this leads to high dislocation density and high strength.[11]

The nanograined materials produced by SPD usually exhibit high elongation to failure levels, which are sometimes comparable to those for coarse-grained materials, and they exhibit uniform ductilities somewhat higher than the uniform ductility of nanograined materials produced by other methods.[1–4] It was suggested that the formation of non-equilibrium grain boundaries,[1] the enhancement of atomic diffusion [37] and activation of other deformation mechanisms such as grain boundary sliding [38] are responsible for such improvements in ductility. Since deformation is localized in SPD-processed materials during tensile test because of low strain-rate sensitivity and/or insufficient capability for further deformation hardening (dislocations are emitted and annihilated at grain boundaries),[39] the uniform ductility of these materials should be improved by using appropriate strategies.

A better combination of high strength and high ductility is achievable in materials with bimodal grain-size distribution when compared to nanograined materials. The nanograins contribute to high strength while the accumulation of dislocations in coarse grains contributes to high ductility.[14–16] Kimura et al. reported that in a low-carbon steel, the presence of elongated grains strengthened by nanometer-sized carbides can result in improvement of both strength and ductility.[17] The current Fe–Ni–Al–C alloy also exhibits similar microstructural feature with capability of accumulation of dislocations in the coarse grains. Poor ductility in the sample processed by HPT for  $N = 50$  should be mainly due to the absence of coarse grains as well as due to the development of tiny cracks.

In summary, an Fe–Ni–Al–C alloy with ultrahigh strength and high ductility was developed in combination with several strategies, namely composition control for lattice softening, SPD processing for grain refinement, control of strain to produce multimodal structure composed of nanograins, elongated coarse grains and fine precipitates.

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