Communication

Strain-Induced Phase Transformation and Nanocrystallization of 301 Metastable Stainless Steel Upon Ultrasonic Shot Peening

KEJIAN LI, KEESAM SHIN, and PENGJUN CAO

The following study investigated the strain-induced phase transformation in metastable austenitic 301 stainless steels *via* an ultrasonic shot peening treatment (USP) for 5 to 30 minutes. Following the USP, the microhardness increased to a depth of 400 μm and from 200 to 400 HV. The deformed grains and the phase transformation were monitored *via* X-ray diffraction and electron backscattered diffraction analysis. The grain evolution was studied *via* transmission electron microscopy. Approximately 500 nm α'-martensite grains formed in the top-most region after 5 minutes of the USP treatment. The grains were then further refined to ~ 100 nm when the peening time increased to 10 and 15 minutes. The grains refined down to tens of nanometers after the specimen was treated for 30 minutes, where the phases were composed of α'-martensite (~ 50 nm). There was a mixture of austenite with α'-martensite (~ 25 nm). The grain refinement and the phase transformation of austenite to α'-martensite during ultrasonic shot peening were systematically investigated.

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Metastable austenitic 301 stainless steels exhibit good ductility and are used in applications that require good formability, such as the formation of sheets, strips, and wires. Following annealing, the soft austenite structure could be obtained that could undergo the martensite transformation during subsequent operations, such as cold working or cryogenic treatments. These steels are not as formable as other conventional stable austenitic stainless steels, although they exhibit a good combination of formability, strength, ductility, and corrosion resistance, making them competitive with other high-strength steels and alloys.[1,2]

The coarse grain refinement of metals and alloys that have dimensions as low as 100 nm could improve the properties of the resulting materials.[3,4] There have been efforts made to refine coarse grains into nanocrystals *via* strain-induced grain refinement using various techniques, including shot peening.[5,6] Ultrasonic shot peening (USP) was used to enhance the surface properties of the metallic parts by inducing phase transformation/microstructural refinement or nanocrystallization that originates from the high strain rate introduced by high energy ultrasonic vibration.[7–10] The mechanical properties of steels with low-stacking fault energy, such as metastable austenite 301 stainless steel, is affected by the strain-induced grain refinement and the phase transformation.

The 301 stainless steel was chemically composed of C 0.05 to 0.15, Si < 2.00, Mn < 2.00, P < 0.045, S < 0.015, N < 0.11, Cr 16.00 to 19.00, Mo < 0.80, and Ni 6.00 to 9.50 (wt. pct). The description of the USP equipment was as previously reported.[9,10] Prior to the treatment, the plate surface was ground to 2000-grit with SiC metallographic paper. The USP treatments were performed at room temperature with an amplitude of 70 μm, frequency of 20 kHz, ball size of 1.5 mm, and a peening area of 2800 mm^2 in the center of a plate (110 × 30 × 5 mm^3).

Following the USP treatment, an analysis of the surface and a cross-sectional region of the specimens were performed *via* X-ray diffraction (XRD), Vickers hardness, electron backscattered diffraction (EBSD), and transmission electron microscopy (TEM). XRD analyses of the untreated specimen and the treated surface layer were performed on a Philips X'pert MPD X-ray (Philips, Netherlands) diffraction with Cu $K_{\alpha 1}$ radiation, with steps of 0.002 deg per second, ranging between 30 and 100 deg. The Vickers hardness test was performed with a load of 200 g and a loading time of 10 seconds. The samples were taken from the treatment surface and the side of the cross section with spacing of indents of around 20 μm. EBSD measurements were performed with a TSL-OIM system installed in a MIRA II LMH FE-SEM in depth beam scan mode, a beam step size of 200 nm, and about 2 hours of scan time for each map. TEM was performed on a Philips CM200 operated at 200 kV.

The cross-sectional specimens for the EBSD analysis were prepared *via* mechanical polishing followed by polishing in colloidal silica for 2 hours. The TEM observation was performed with back-thinning

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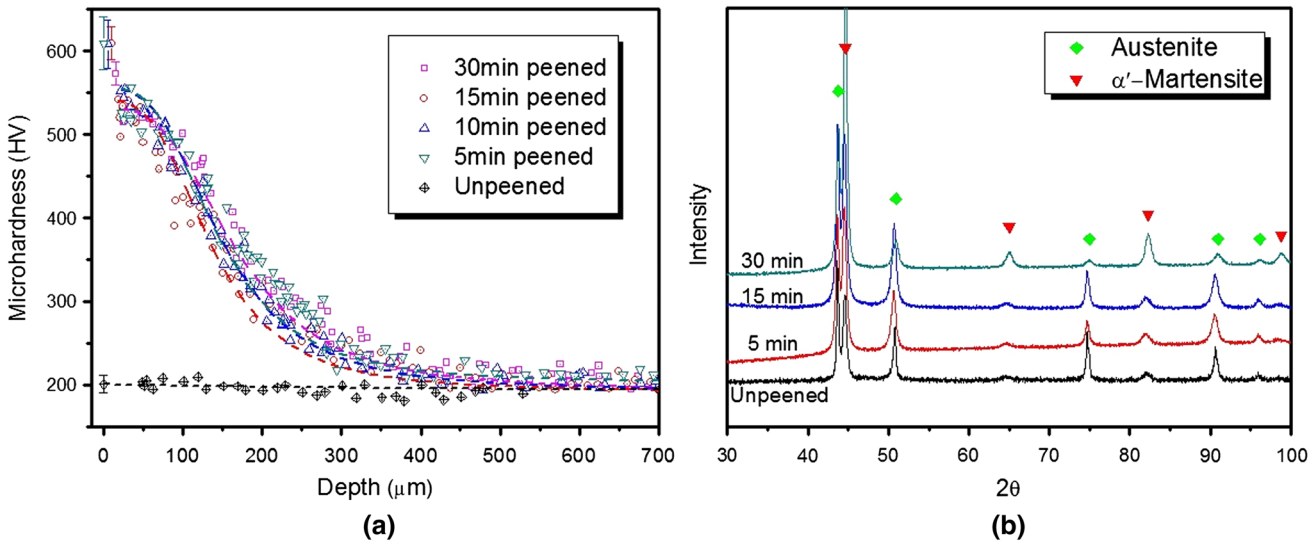

Fig. 1—Vickers hardness (*a*) and X-ray diffraction (*b*) profiles of specimens before and after the USP treatment.

processing to prepare the top-most surface layer. The top treated surface specimens were thinned *via* polishing to 90 μm from the side opposite to the treated side. They were then attached to a transparent plastic film on the treated side to prevent etching/polishing during electrolytic polishing. The TEM specimens were electro-polished (Struers TenuPol-5) at room temperature with an electrolyte mixture of 10 pct perchloric and 90 pct acetic acid.

The effect that USP had on microhardness was determined by cross-sectional measurements. The microhardness profiles of the unpeened and the USP-treated specimens are shown in Figure 1(a). Following USP, the hardness increased to a depth of ~ 400 μm. The maximum was ~ 600 HV at the top surface of the USP-treated sample. This was three times the hardness of the unpeened specimen, which exhibited an average value of ~ 200 HV. The 5- to 15-minute peened specimens exhibited a hardness of ~ 600 HV on the treated surface, where the 30-minute peened specimens exhibited a hardness of ~ 575 HV. The error in the hardness measurement decreased as peening time increased on the treatment surface.

Figure 1(b) shows the XRD patterns of the unpeened and the USP-treated specimens. Two phases (austenite and α'-martensite) were present in the XRD peaks, indicating that the α'-martensite increased after the USP treatment.

Figure 2 shows the cross-sectional EBSD phase mapping images of the (a) unpeened, (b) after 5 minutes, (c) after 10 minutes, (d) after 15 minutes, and (e) after 30 minutes of USP treatment. The green color indicated the austenite phase and the red color indicated the α'-martensite phase. In the unpeened specimens, nearly all the matrix was austenite and annealing twins were present in the coarse grains. Following 5 minutes of the USP treatment, the deformed slip band structures became obvious in the original grains and the α'-martensite followed the slip band structures, as shown in (b). The slip bands increased (c) in the specimens that received 10 minutes of USP treatment. As the peening time increased to 15 minutes, a higher density of the α'-martensite phase and the grain refinement was present, as shown in (d). For samples treated with 30 minutes of the USP treatment, nearly all the grains were the α'-martensite phase near the top treated surface. The red color became predominant, signifying the progressive increase of the α'-martensite phase in the USP-affected region, which was consistent with the results from the XRD analysis.

Figure 3(a) shows a higher magnification of the EBSD mapping, focused on the rectangular area of Figure 2(d). A well-arranged lath-shaped α'-martensite was apparent at 20 to 50 μm with the 15-minute peened specimen. After high-angle grain boundaries were added (15 to 180 deg), the refined grains appeared as shown in Figure 3(b). TEM sampling was taken at a depth of ~ 30 μm in this area. The bright field and the dark field TEM micrograph shows the lath-shaped grains (width ~ 500 nm) from two directions at an angle of ~ 75 deg. The inset displaying the selected area diffraction pattern (SADP) confirmed the α'-martensite phase. The dark field image used the reflection of the (110) spots of the first ring in the SADP.

TEM analyses revealed the grain refinement and the phase transformation after USP treatment. The TEM foils were prepared at the top-most layers of the peened specimens, as shown in Figure 4, which showed the bright field images in (a_1 to c_1), dark field images in (a_2 to c_2), and corresponding SADPs in (a_3 to c_3). The dark field image used the reflection of the first ring spots. In the 5-minute peened specimen ($a_{1\sim3}$), the grains were lath shaped with slips. The α'-martensite phase, with a mean width of ~ 200 nm and mean length of ~ 800 nm, was similar to the structures in the sample that was peened for 15 minutes at ~ 30 μm depth. The SADPs exhibited almost complete ring patterns (b_3, c_3), indicating that the number density of the nanoscale grains was fairly high. This suggested the significance of the surface nanocrystallization on the surface of the 301 stainless steel.

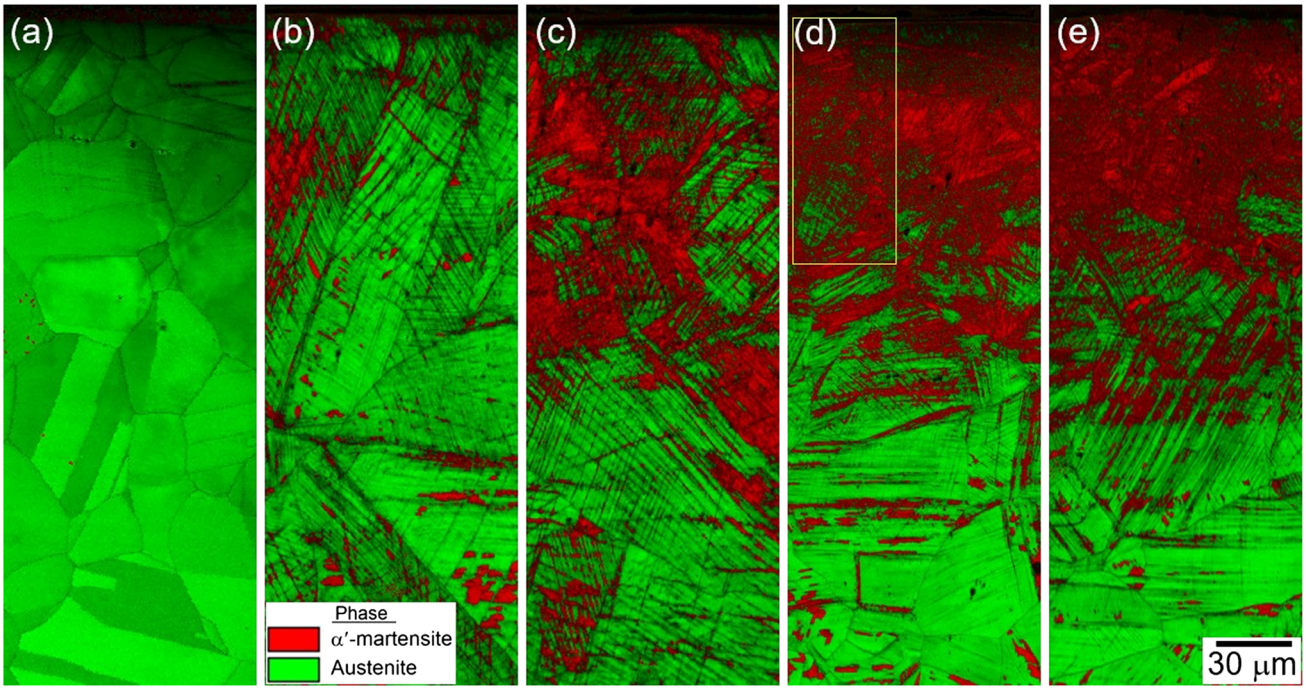

Fig. 2—Cross-sectional EBSD phase mapping of (*a*) the unpeened, (*b*) after 5 min, (*c*) after 10 min, (*d*) after 15 min, and (*e*) after 30 min of USP treatment.

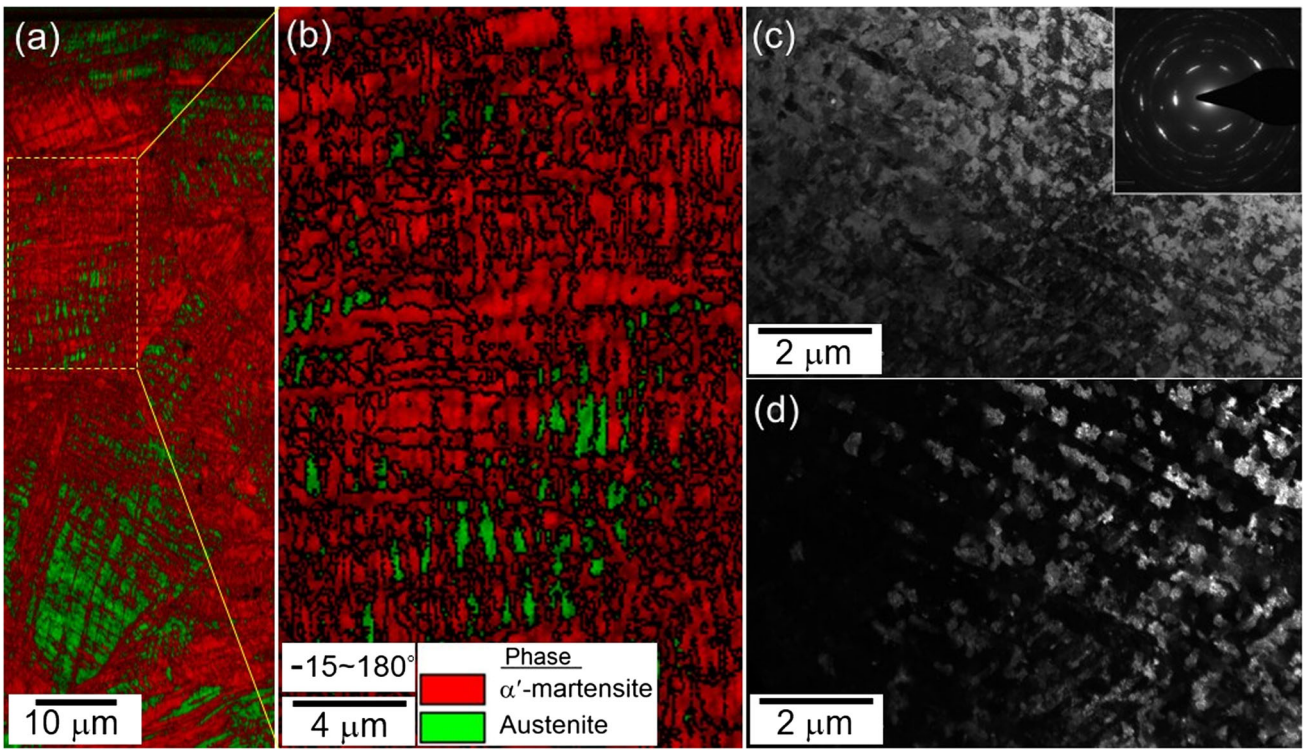

Fig. 3—EBSD and TEM analysis of the 15-min peened specimen: (*a*, *b*) EBSD phase mapping at a high magnification of the rectangular region of Fig. 2(d), TEM (*c*) bright field, and (*d*) dark field images taken at ~ 30 μm depth. The inset was SADP.

Figure 5 shows the TEM analysis in the top-most layer of the 30-minute peened specimen. The TEM bright field image showed the nanoscale grains in (a); however, there was a demarcation line in the nanoscale grains, which was marked by a yellow dotted line. There were different phases in the left and right regions. The yellow dotted line was marked on the interface of the two phases. Two SADPs were obtained in this region,

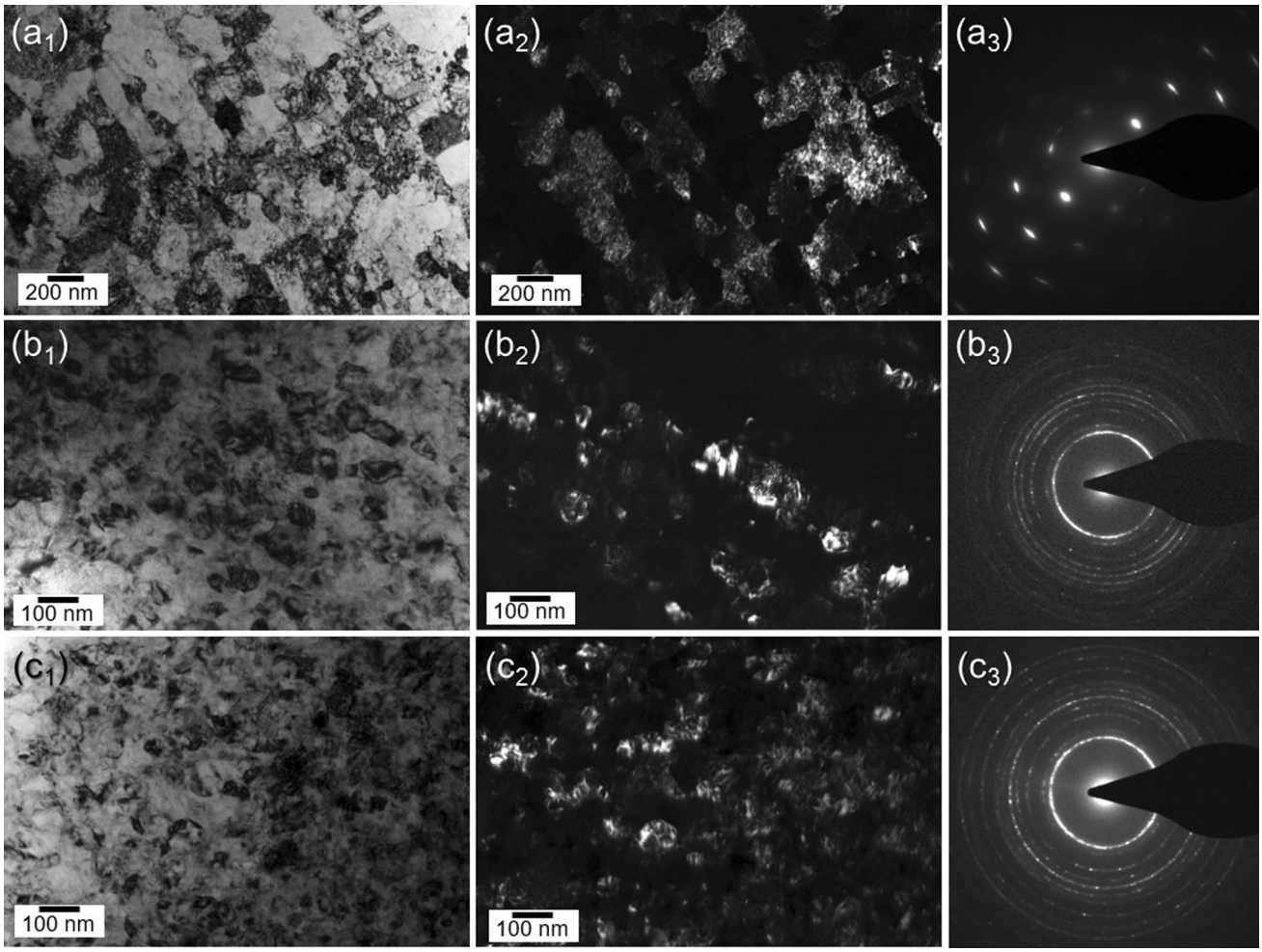

Fig. 4—TEM images at top-most layer of ($a_{1–3}$) after 5 min, ($b_{1–3}$) after 10 min, and ($c_{1–3}$) after 15 min of USP treatment. Grain refinement was observed between 5 and 15 min, the surface nanocrystallizations were confirmed in the 10- and 15-min peened specimens of the 301 stainless steel.

revealing that the left part was the α′-martensite and the right part was a mixture of the α′-martensite and the austenite. The average grain size of the left part was 50 nm and right part was 25 nm. A grain size measurement was taken with Image-Pro plus software. The high-resolution TEM image was taken for the 30-minute peened specimen and shown in (b). The fast Fourier transform (FFT) images from the dotted square region are shown in (c). A real line square region is shown in (d). (c) was taken from the [110] direction face-centered cubic (FCC) crystal structure of austenite for the pattern analyses and (d) was taken from the [111] direction body-centered cubic (BCC) of the α′-martensite. Corresponding inverse FFT images of the high-resolution TEM image that showed the austenite and the α′-martensite atomic positions are shown in (e) and (f).

Vickers hardness of the 5-minute peened specimens had the quickest hardness increase, which means the efficiency is higher in 5 minutes than after. Due to the measurement of the phase fraction *via* EBSD inverse pole figure (IPF) maps, an approximate 50-50 volume ratio of the austenite and the α′-martensite would provide good corrosion resistance for many duplex phase stainless steels.[11–13] The volume ratio of austenite to the α′-martensite phase is 54:46 upon three times measurement on different field in the 10-minute peened specimens, demonstrating that the 10-minute peened specimen might be the best peening parameter for industrial applications. The hardness for the 30-minute specimens was less than the 5- to 15-minute peened specimens. The specimens peened between 5 and 15 minutes were regarded as the thermal equilibrium with dynamic recrystallization during the long time USP treatment, where grain growth and refinement concurrently occurred near the top treatment surface.[9]

The austenite to the α′-martensite phase transformation during USP treatment was apparent in the XRD and EBSD results (Figures 1(b) and 2). The formation of the α′-martensite followed the slip bands and the deformation twins. As the peening time increased, the grain refinement continued as the α′-martensite increased. The well-arranged lath-shaped α′-martensite

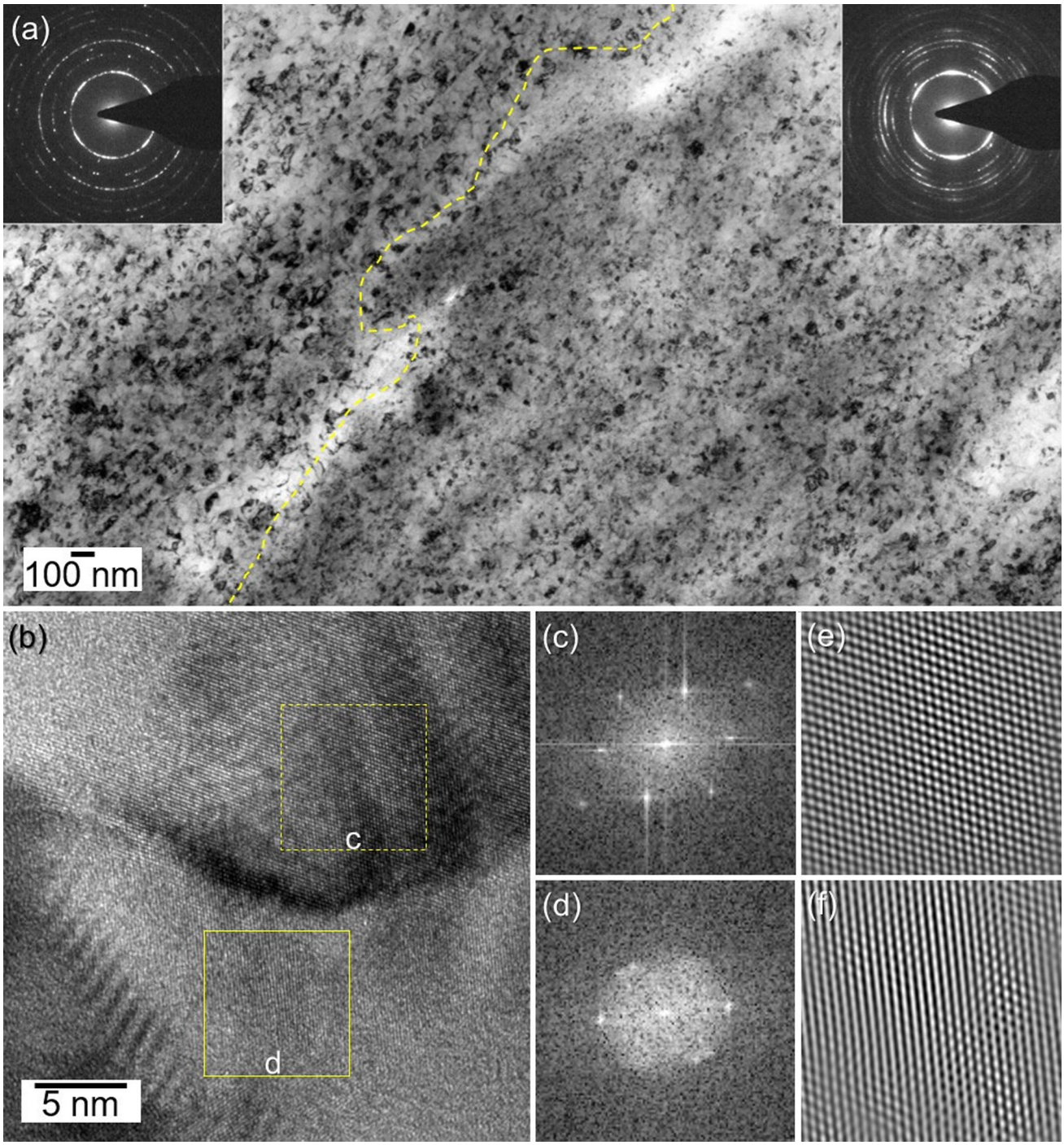

Fig. 5—TEM images at the upper layer of the 30-min USP-treated specimen. (*a*) Bright field image with inset of the SADPs in the left and right regions, (*b*) a high-resolution TEM image, (*c*, *d*) FFT images taken from the square region, and (*e*, *f*) corresponding inverse FFT images.

in Figures 3(b) and (c) showed that nearly all austenite transformed to the α'-martensite phase. The observation of the ultra-fine grains in the 10- to 30-minute treated specimen suggested that severe strain induced the grain refinement of the austenite, the α'-martensite, and the nanoscale grains, including two phases on the peening surface.

In this study, a 301 metastable austenite stainless steel was USP treated for 5 to 30 minutes. The conclusions for the microhardness and the microstructure evolution were as follows:

1. The hardness on the top-most layer obtained a three-time increase from 200 to 600 HV. EBSD analysis indicated the grain refinement and deformation structures, the influence depth of hardness increase and formation of deformed structure is 400 μm after USP treatment.

2. The formation of the α′-martensite followed the slip band and the deformation twins after USP treatment. As the peening time increased, the refinement of deformed grains continued as the α′-martensite increased.
3. In the 5-minute USP-treated specimen, the α′-martensite grains were formed in the high deformation region forming a lath shape, with a mean width of ~ 200 nm. The mean length was ~ 800 nm. The mean grain size was further refined to ~ 100 nm as the peening time increased to 10 and 15 minutes.
4. The grains were refined to a nanoscale size in the 30-minute treated specimens. The phases were composed of the α′-martensite (~ 50 nm in size) with a mixture of austenite and α′-martensite (~ 25 nm in size).

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