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Work hardening behavior of nanotwinned austenitic grains in a metastable austenitic stainless steel

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Work hardening capability is the key factor to determine the ductility and toughness of materials. For the metastable stainless steel (SS) with low stacking fault energy (SFE), work hardening rate increases again with straining after the monotonous decay in the Kocks-stage [\[1,2\]](#page--1-0). Such an abnormal multistage behavior of work hardening is mainly ascribed to strain induced martensitic transformation (SIMT), thereby resulting in an excellent ductility, i.e. transformation induced plasticity (TRIP) effect [\[3,4\].](#page--1-0) However, many investigations indicated that when the size of austenitic grains was refined to nanoscale, SIMT was suppressed, and then a Kocks–Mecking type behavior instead of the abnormal work hardening was observed [\[5,6\].](#page--1-0) Consequently, the nanostructured austenitic stainless steels exhibited a very limited ductility at a high strength. For example, the uniform elongation is very low for the nanostructured 304L SS with grain sizes below 500 nm at the yield strength of 900 MPa [\[7\]](#page--1-0).

Recently, a novel strategy is proposed for strengthening austenitic steels with considerable work hardening capability by introducing nanotwinned austenite (nt-γ) grains which contain multiple twins with twin boundaries (TB) spaced in the nanometer regime [8-[13\].](#page--1-0) This new type of nanotwinned steels exhibited a good ductility at a high strength. Our recent work showed that the nanotwinned 304 SS exhibits a uniform elongation of \sim 13% with the yield strength of as high as ~1.0 GPa [\[14\].](#page--1-0) The $nt-\gamma$ grains play a key role in the strength and plasticity, which is closely related to the work hardening of the nanotwinned 304 SS. In the present work, we investigated the work hardening behavior of nt-γ grains by means of ex-situ tensile tests.

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A commercial AISI 304 SS with a composition of Fe–18.46Cr–8.28Ni– 0.012Mo–0.049C–0.42Si–1.64Mn–0.003S–0.021P (wt.%) was used in this work. The as-received samples were annealed at 1473 K for 2 h followed by air cooling to obtain austenitic coarse grains (averagely \sim 140 μm). The cylindrical samples were processed by using dynamic plastic deformation (DPD) at 423 K with $\varepsilon = 1.0$ to introduce numerous nt-γ grains. The DPD set up and processing parameters were described elsewhere [\[15\]](#page--1-0). The DPD sample (hereafter referred to as nanotwinned 304 SS) was then annealed at 923 K for different durations to obtain various microstructures.

The work hardening behavior of nanotwinned austenitic grains in a novel type of nanotwinned austenitic stainless steels was investigated. Multiple Shockley partial dislocation activities in the nanotwinned grains could trigger occurrence of stacking faults and strain induced martensitic transformation before necking, resulting in an increase in work hardening rate. Consequently, the fully recovered nanotwinned austenitic

stainless steel exhibits a uniform elongation of ~13% at the strength of 950 MPa.

Microstructures were characterized by scanning electron microscope FEI Nova NanoSEM 430 with electron channeling contrast (ECC) imaging and a transmission electron microscope JEOL 2010 operated at 200 kV. Previous results revealed that the nanotwinned 304 SS consists of ~58 vol.% nt-γ grains and ~42 vol.% dislocation structures [\[14\]](#page--1-0). When annealed at 923 K for 15 min, the microstructure is nearly unchanged except the reduction of dislocation density (hereafter referred as to partially recovered nanotwinned sample). With further annealing to 45 min, as shown in [Fig. 1a](#page-1-0), numerous parallel strips and indistinct regions as well as a negligible amount of SRX grains were observed in this sample (hereafter referred as to fully recovered nanotwinned sample) from the SEM image. Detailed TEM observations verified that the parallel strips are high density of nano-scale twins and the indistinct region consists of dislocation structures ([Fig. 1b](#page-1-0)–c). The mixed structure consists of ~58 vol.% nt- γ grains, ~40 vol.% dislocation structures and ~2 vol.% static recrystallized (SRX) grains. The twin/matrix (T/M) lamella thickness is in the range from a few to \sim 100 nm with an average value of \sim 19 nm. The contrast of the T/M lamellae is relatively uniform and the TBs are straight and relatively clean, which indicates a low dislocation density of the nt-γ grains. On

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Fig. 1. Typical microstructures of the annealed nanotwinned 304 SS at 923 K for 45 min: (a) SEM-ECC image; TEM image of (b) the nt-γ grains and (c) dislocation structures with SAED patterns (inset). DS: dislocation structures.

the other hand, the dislocation structures consist of dislocation tangles, walls and cells, which are analogous to that observed in the deformed 304 SS [\[16\]](#page--1-0). The X-ray diffraction (XRD) results show that the annealed nanotwinned sample consists of a single austenitic phase.

Uniaxial tensile tests were performed on an Instron 5848 Micro-Tester system with a strain rate of 5×10^{-3} at room temperature. A contactless MTS LX 300 laser extensometer was used to measure strain in the sample gage upon loading. The tensile specimens were cut into a dog-bone shape with a gage section of $5 \times 1 \times 0.5$ mm³. The nanotwinned sample exhibited a high tensile strength of \sim 1135 \pm 79 MPa without uniform ductility [\[17\].](#page--1-0) After thermal annealing, its ductility increases accompanied by the decrease of the strength, consistent with other deformed and annealed materials [\[9,10\],](#page--1-0) as shown in Fig. 2a. Note that the partially recovered nanotwinned sample exhibits a tensile uniform elongation of \sim 3.4 \pm 0.1% while for the fully recovered nanotwinned sample, the uniform elongation is as high as \sim 13 \pm 1%, about four times higher than that of the former at the yield strength of 950 \pm 8 MPa. This high abnormal ductility is mainly ascribed to its excellent work hardening behavior.

The work hardening ($\Theta = d\sigma / d\varepsilon$) of the two recovered nanotwinned 304 samples is characterized by abnormal multistage behaviors, as shown in Fig. 2b. Clearly, after a steep decrease in work hardening for the elastic–plastic transition, the work hardening rates of both samples exhibit an identical linear decrease to ~900 MPa at intermediate strains (first stage), which is similar to the classical stage III hardening regime of copper and aluminum [\[18\].](#page--1-0) With further straining, the sharp drop of work hardening is suppressed and then the work hardening maintains at a constant value for the partially recovered sample and increases slightly for the fully recovered one (second stage). For example, the work hardening rate of the fully recovered nanotwinned sample increases from ~900 MPa to ~1120 MPa in the strain range from 6% to 14%.

In order to analyze the special work hardening behavior of $nt-\gamma$ grains, the microstructures of the fully recovered nanotwinned samples at various strains (4% and 10%) were characterized by TEM and XRD analyses. TEM observations, as expected, revealed that no obvious change was observed in the ~40 vol.% dislocation structures with straining. This is consistent with the previous work that the ultrafine dislocation structures usually present a sharp drop of work hardening over a small plastic strain of 1–2% [\[12\]](#page--1-0). In addition, the volume fraction of the SRX grains is negligible in this sample. So the nt - γ grains may play a dominate role in enhancing the work hardening rate for the fully recovered nanotwinned sample. The following analyses are focused on the microstructural evolution of the $nt-\gamma$ grains during tensile test.

Fig. 2. (a) Tensile true stress–strain curves of the partially and fully recovered nanotwinned 304 SS; (b) Work hardening rate Θ vs. true strain for the fully recovered nanotwinned austenite 304 SS compared with that for the partially recovered nanotwinned 304 SS. The cross symbols indicate the uniform elongation for both samples.

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