Contents lists available at ScienceDirect

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Heterogeneous nanostructure developed in heavily cold-rolled stainless steels and the specific mechanical properties



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ARTICLE INFO

Article history: Received 27 January 2017 Received in revised form 7 February 2017 Accepted 8 February 2017 Available online xxxx

Keywords: Heterogeneous nanostructure Twin Ultrafine grain Rolling Mechanical property

ABSTRACT

Stainless steels were cold rolled to 92% reduction. The microstructure developed in a stable austenitic stainless steel was complicated heterogeneous nanostructure composed of lamellar-twin domains, shear bands and low-angle lamellar boundaries. Cold-rolled duplex stainless steel, however, exhibited more complicated and mixed microstructure where ferritic lamellar and the heterogeneous nanostructure of austenite were alternatively stacked. The heavily cold-rolled stainless steels followed by ageing exhibited marvelous high tensile strength over 2.6 GPa perpendicular to the rolling direction, although around 2 GPa along the rolling direction. Even while such high strength, moderate plastic elongation over 5% was attained.

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Various methods for severe plastic deformation (SPD) are proposed to attain ultrafine-grained (UFGed) metallic materials [1–7]. It is being revealed that the UFGed materials possess various specific properties as well as high strength. The sample sizes of the UFGed materials processed by SPD methods are, however, generally too small to employ as structural elements. Still more, the most of SPD methods are the socalled batch processes with low efficiency and complicated to employ for factory mass production. New methods for fabrication of bulky UFGed metallic materials suitable for mass production are therefore highly desirable.

The minimum grain size achieved by SPD methods appears roughly around 0.1–0.2 µm independent of processes [1]. This size is comparable with that of subgrains. This fact would imply that the most dominant grain-subdivision mechanism operative during SPD is strain-induced grain refinement related with dislocation accumulation and gradual evolution of substructure [3]. However, the grain refinement is dramatically accelerated and much finer grain-sized microstructure can be fabricated when mechanical twinning is involved in grain refinement process [6]. This is because that mechanical twinning with boundary spacing of about 20 nm contributes to drastic and instant subdivisions of coarse initial grains. Actually, a homogeneous UFGed structure with average grain size of 20 nm was successfully produced by multi-directional forging (MDF) of SUS316L austenitic stainless steel and Cu—Zn

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alloy due to mechanical twinning [6,7]. It was also found that grain subdivision by multiple mechanical twinning and UFG evolution took place even by heavy cold rolling of low stacking-fault-energy (SFE) Cu—Be alloy and ultrahigh tensile strength of 1.8 GPa was achieved after ageing [8]. In the latter case, the conventional lamellar and primary twins developed by cold rolling were further subdivided by mechanical twinning to form UFGed structure. The above results suggest that formation of UFGed structure would be also possible in stable austenitic stainless steels with low SFE by simple heavy cold rolling when dense formation of mechanical twinning takes place. If it is, UFGed stainless steel sheets with ultra high strength can be mass producible.

SUS316LN and SUS310S stable austenitic stainless steels and DIN1.4462(SUS329J3L) duplex stainless steel were solution treated at 1523 K for 1 h followed by water quenching. They were subsequently 92% cold rolled to have thickness of 0.5 mm. Here after, these samples will be simply referred as SUS316LN, SUS310S and DIN1.4462, respectively. The volume fraction of austenite (γ) phase in the samples after cold rolling measured by the ferrite content meter was 100%, 100% and 70%, respectively. No martensitic transformation was detected in the stable austenitic stainless steels even after heavy cold rolling. The cold-rolled samples were aged at 773 K from 10 s to 604,800 s (168 h) in the salt bath. The evolved microstructure was investigated by means of orientation imaging microscopy (OIM) and transmission electron microscopy (TEM) after mechanical and electritical polishing. For the TEM observations from transvers direction (T.D.), foils were prepared by an ion thinning method. Mechanical properties were

http://dx.doi.org/10.1016/j.scriptamat.2017.02.016

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Fig. 1. Change in hardness during ageing at 773 K of SUS316LN, SUS310S austenitic and DIN1.4462 duplex stainless steels. Results obtained by multi-directional forging (MDFing) of SUS316L stainless steel at 300 K and 77 K were also shown for comparison [6].

characterized using micro-Vickers hardness tester and Instron-type mechanical testing machine. The tensile specimens with gauge dimensions of $6 \times 2.25 \times 0.5$ mm³ were electro-discharge machined. Tensile test was carried out parallel to rolling direction (R.D.) and T.D. at an initial strain rate of 2.5×10^{-3} s⁻¹ at room temperature.

Fig. 1 shows change in hardness during ageing at 773 K of the 92% cold-rolled samples. The hardness of the cold-rolled samples was almost comparable to those attained in SUS316L stainless steels MDFed at 77 K and 300 K to cumulative strain of 6.0. The hardness gradually

increased by ageing and quite high peak hardness over 5 GPa could be achieved in all the samples, while it of the SUS310S was relatively lower. The large age-hardenability should be affected by good thermal stability of the microstructures introduced by heavy cold rolling and relatively low ageing temperature in addition to the intrinsic agehardenability. It is known that UFGed stainless steel possesses quite low thermal stability to cause grain growth at temperatures as low as 823 K and, therefore, low age-hardenability [9]. Here after, investigation is focused mainly on SUS316LN and DIN1.4462 because of their extremely high hardness.

The typical microstructures evolved after cold rolling are displayed in Fig. 2. In the SUS316LN, thin lamellar structure with average width of 29.5 nm and almost parallel to R.D. can be seen. What notable in Fig. 2 (a) is that "eye" shaped domain, which would be described precisely in the next paragraph, was embedded in the conventional lamellar to form complicated nanostructure. The "eye" domain itself was also composed of lamellar. On the other hand, the microstructure developed in the DIN1.4462 looks much more intricate. In the α phase region, a typical and conventional lamellar with dislocation substructure could be observed to develop. Whereas, in the γ phase regions, much more complicated lamellar structure with embedded "eye" domain can be seen. However, the size of "eye" domain in γ phase was relatively larger and not densely distributed compared with that in the SUS316LN.

The "eye" domain developed in the SUS316LN was more precisely investigated and the further magnified image is exhibited in Fig. 3. It was confirmed from the analyses of selected-area-diffraction pattern and OIM, the "eye" domain is mainly composed of thin lamellar twins, in which some part of the lamellar twins were further subdivided by higher order twins. The primary twin boundary spacing in the "eye" domain was about 37 nm. The "eye" domain was surrounded by shear bands and was embedded in the conventional lamellar structure. Because all the component microstructures are those of deformation induced, the complicated microstructure evolved by heavy cold rolling would be called as "heterogeneous" nanostructure. After peak ageing, the lamellar spacing became wider to 42.3 nm in average (Fig. 3(b)).



Fig. 2. TEM micrographs observed in (a) SUS316LN, (b) and (c) DIN1.4462 duplex stainless steels. (b) and (c) are the characteristic microstructures developed in α and γ phases respectively in the DIN1.4462 duplex stainless steel. The corresponding schematic illustrations of the microstructures are exhibited in (d) and (e). R.D. and N.D. indicate rolling and normal directions, respectively.

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