Materials Science and Engineering A 528 (2011) 1310–1317

Contents lists available at ScienceDirect

Materials Science and Engineering A

journal homepage: www.elsevier.com/locate/msea

# Nano-grain evolution in austenitic stainless steel during multi-directional forging

Y. Nakao, H. Miura*

*Department of Mechanical Engineering and Intelligent Systems, UEC Tokyo (The University of Electro-Communications), Chofu, Tokyo 182-8585, Japan*

ARTICLE INFO

*Article history:*
Received 27 May 2010
Received in revised form 6 October 2010
Accepted 9 October 2010

*Keywords:*
Ultrafine grained microstructure
Severe plastic deformation
Twinning
Multi-directional forging
Strain-induced martensitic transformation

ABSTRACT

Nano-grain evolution in an austenitic stainless steel (SUS 316) during multidirectional forging (MDF) was investigated at temperatures of 77 K and 300 K. The flow stress during MDF and the room-temperature hardness increased significantly with increasing cumulative strain. The initial grains were subdivided by mechanical twinning and martensitic transformation. The formation of *packets*, which are composed of lamellar-structured mechanical twins with a spacing of 10–300 nm, enhanced grain fragmentation. The packet size ranged from 40 nm to 100 nm depending on the MDF temperature and the cumulative strain. Tensile tests at ambient temperatures revealed a maximum proof strength of 2.1 GPa. While the proof strength increased with cumulative strain, the plastic strain at fracture was approximately 10% independent of the cumulative strain over $\sum \Delta\varepsilon = 2.4$.

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## 1. Introduction

The well-known Hall–Petch relation states that the strength of metallic materials increases with a reduction in the grain size. Although numerous studies have been carried out to reduce the grain size using conventional thermo-mechanical processing, it is quite difficult to obtain a homogeneous 1 μm grain structure. Therefore, grain refinement using severe plastic deformation (SPD) has been studied. Various SPD methods, such as equal-channel angular pressing (ECAP) [1–4], accumulative roll bonding (ARB) [5–7], and multidirectional forging (MDF) [8–11] have been successfully applied to obtain ultrafine grains (UFGs) in bulky metallic materials.

Belyakov et al. and Takayama et al. explained that the mechanism of UFG evolution during MDF is similar to continuous dynamic recrystallization (cDRX) [9,10]. In this mechanism, the strain-induced UFGs are the result of dislocation generation and the formation of high-angle grain boundaries by accumulation onto the dislocation walls, subgrain boundaries, and low angle grain boundaries. The minimum grain size obtained by this mechanism appears to be 0.1–0.3 μm, which is the size of the subgrains. However, further grain refinement remains difficult.

Miura et al. reported that grain refinement to a mean size of 20 nm was achieved by MDF at 77 K in a Cu–Zn alloy [12]. They found that the grain refinement was enhanced by grain subdivision using mechanical twinning. It is known that mechanical twins evolve easily at cryogenic temperatures in the metallic materials that possess low stacking-fault energies [13,14]. Because the twin boundary, as well as the general grain boundaries, contribute to strengthening [15–17], UFGed materials fragmented by mechanical twinning exhibit high strength [18].

Strain-induced martensitic transformation contributes to grain fragmentation [19,20]. The lamellar spacing of strain-induced martensites is as fine as that of mechanical twins [19,21]. The strain-induced martensite appears more easily and frequently with increasing strain and/or decreasing deformation temperature in austenitic stainless steels [22]. It is therefore suggested that grain refinement in certain materials can be enhanced by a synergetic mechanism involving cDRX, mechanical twinning, and martensitic transformation.

Many studies of the mechanical properties of SPDed materials have been carried out. It has been revealed that UFGed materials ($d \geq 0.2$ μm) possess high strength but relatively poor ductility [23–25]. One explanation for these mechanical properties is the rather short mean free path of the dislocations. However, it has also been shown that a Cu–Zn alloy composed of 20 nm grains exhibits rather good ductility [18]. This implies that ductility may be recovered by the evolution of UFGs much smaller than 0.1 μm and by control of the grain-boundary character distribution.

The goal of the present study is to investigate UFG evolution in austenitic stainless steel during MDF using the coordinated mechanisms of mechanical twinning, martensitic transformation, and cDRX. The mechanical properties of the UFGed stainless steel are also studied.

* Corresponding author.
*E-mail address:* miura@mce.uec.ac.jp (H. Miura).

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doi:10.1016/j.msea.2010.10.018

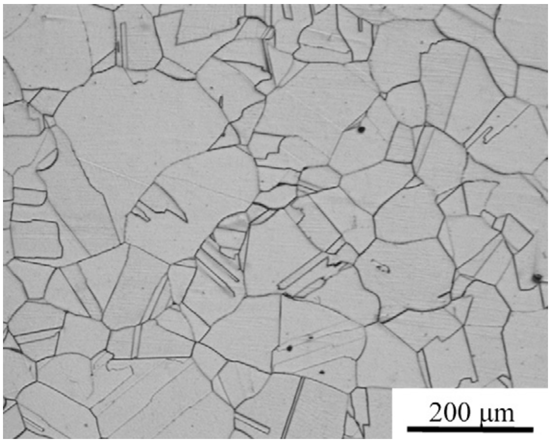


**Fig. 1.** Initial microstructure of 316-type stainless steel obtained by annealing at 1073 K for 30 min.

## 2. Experimental procedure

A rectangular 316-type austenitic stainless steel (Fe–16.9Cr–10.05Ni–2.04Mo–1.37Mn–0.044C–0.35Si–0.33Cu–0.03P–0.03S (in mass%)) sample with dimensions 10 mm × 12.2 mm × 15 mm was annealed at 1073 K for 30 min to obtain an average grain size of 100 μm (Fig. 1). MDF was subsequently performed to a maximum cumulative strain of $\sum\Delta\varepsilon = 6$. The samples were MDFed at 77 K and 300 K on an Amsler-type mechanical testing machine at an initial strain rate of approximately $3 \times 10^{-3}\ s^{-1}$. In the MDF method, the samples were compressed with a changing loading direction of 90° pass-by-pass (i.e., *z* to *y* to *x* to *z*. . .) (Fig. 2). The aspect ratio of the samples was almost unchanged during repeated MDF with a pass strain of $\Delta\varepsilon = 0.4$. After MDF, the hardness was measured at room temperature and the arithmetic mean values of 10 data points were plotted.

Microstructural observation was carried out on the sections parallel to the final forging axis (F.A.) using optical microscopy and transmission electron microscopy (TEM). The average grain size or "*packet*" size was estimated by the line-intercept method. For this purpose, 10 photographs at least were used for the evaluation of each data point. The packet is described in a later section. The selected area diffraction (SAD) patterns corresponding to the bright field images were taken using an aperture of 1.3 μm in diameter. The qualitative volume increase in martensitic transformation $\gamma \rightarrow \alpha'$ was estimated using a magnetic method [26]. The MDFed samples were held at a constant magnetic field and the pull power of the sample was measured as 'magnetic force'.

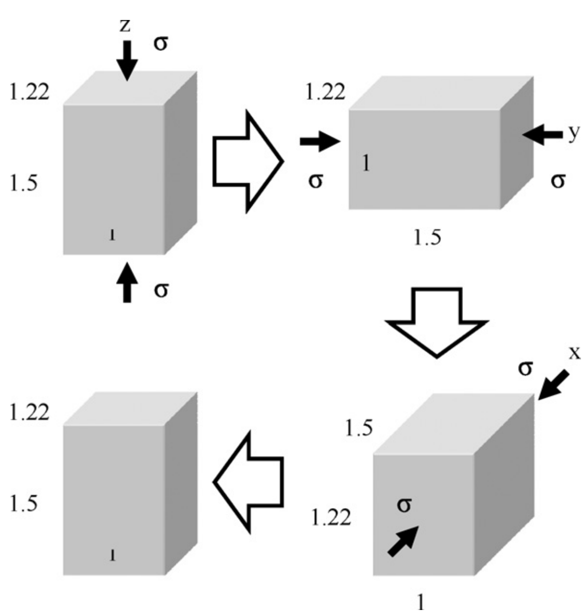


**Fig. 2.** Schematic illustration of the process of multidirectional forging (MDF) with loading stress $\sigma$. The indexed numbers give the aspect ratio of the sample.

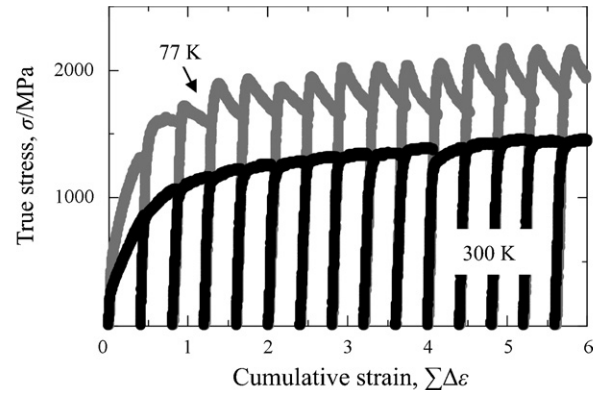


**Fig. 3.** True stress–cumulative strain curves during MDF of an SUS 316 stainless steel at 77 K and 300 K.

A tensile test was carried out using an Instron-type mechanical test set-up at an initial strain rate of $2.5 \times 10^{-3}\ s^{-1}$ at room temperature. The tensile samples with gage dimensions $2 \times 4 \times 0.5\ mm^3$ were discharge machined so that the tensile axis was normal to the final F.A.

## 3. Results and discussion

### 3.1. Flow curves and change in hardness during MDF

The true stress vs. cumulative strain curves obtained during the MDF are shown in Fig. 3. Rapid work hardening occurred in the low-strain region up to $\sum\Delta\varepsilon = 1$. MDF at 77 K exhibited more significant hardening than MDF at 300 K. At 77 K, work softening appeared after the peak stresses for each pass. It is known that work softening is induced by the occurrence of mechanical twinning due to the mitigation of internal stress [13] in addition to the temperature rise during the deformation. However, the temperature rise in a small sample during MDF at such a low strain rate of $3 \times 10^{-3}\ s^{-1}$ is quite

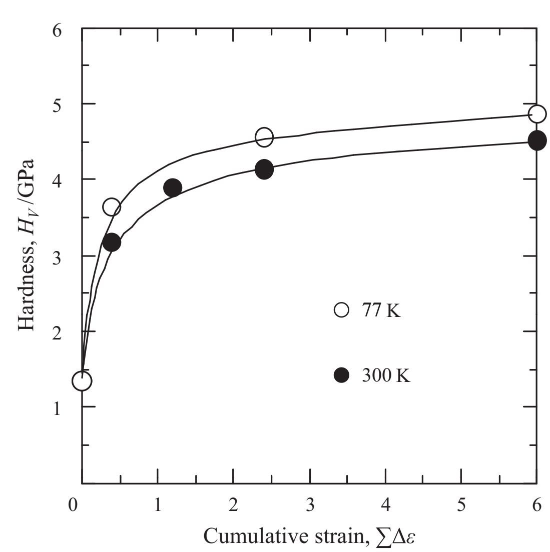


**Fig. 4.** Change in room-temperature Vickers hardness during MDF at 77 K and 300 K.

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