Microstructural evolution of metastable austenitic steel during high-pressure torsion and subsequent heat treatment

S Chen, A Shibata, L J Zhao, S Gao, Y Z Tian and N Tsuji
Department of Materials Science and Engineering, Kyoto University, Yoshida-honmachi, Sakyo-ku, Kyoto 606-8501, Japan
E-mail: chen.shuai.84v@st.kyoto-u.ac.jp

Abstract. Metastable austenite in a Fe-24Ni-0.3C (wt.%) alloy was processed by high-pressure torsion and subsequently heat-treated. The HPT-processed material had lamellae structures composed of highly deformed austenite and deformation-induced martensite. The reverse transformation of the deformation-induced martensite and recovery/recrystallization of the retained austenite completed above 500 °C and resulted in fully annealed and single-phase austenite with different grain sizes. The ultrafine-grained and nanocrystalline austenite showed high yield strength and large ductility due to transformation-induced plasticity.

1. Introduction
Ultrafine-grained (UFG) and nanocrystalline (NC) materials have been a hot topic in the field of metallic materials since last decades. Bulk UFG and NC materials could be fabricated by severe plastic deformation (SPD) methods such as accumulative roll bonding [1, 2] and high-pressure torsion (HPT) [3]. These materials usually have a high density of dislocations and show morphologies of deformed microstructures, and some of them even have unclear boundaries [3]. The UFG materials fabricated by SPD usually show much higher yield strength but lower tensile ductility than those of coarse-grained materials, which is attributed to early plastic instability [1, 4]. Deformation-induced martensitic transformation is expected as one of the ways to suppress the early plastic instability and to enhance the tensile ductility of UFG and NC materials through transformation during tensile deformation [5].

A combination of deformation-induced martensitic transformation and following reverse transformation is also regarded as an effective way among the methods to refine the structure of metastable austenite in steels. The minimum average grain size of fully annealed austenite obtained in this route using cold rolling and subsequent heat treatment was still above 1 μm [6, 7], which might be due to the limited strain by cold rolling. In a recent work by the present authors, larger strain by HPT enabled to obtain UFG and NC metastable austenite with fully annealed structure (minimum mean grain size of 400 nm) in a Fe-24Ni-0.3C alloy (wt.%) [8]. However, the structural evolution during the HPT processing and subsequent heat treatment was not clarified yet. This study is aimed to clarify the structure evolution during HPT processing and subsequent heat treatment and to investigate the mechanical property of the fully annealed UFG austenite.

2. Experiment and methods
An Fe-24Ni-0.3C (wt.%) alloy was annealed at 1173 K for 1 hour to obtain fully recrystallized metastable austenite as the starting material. Samples with dimensions of 10.0 mm in diameter and 0.85 mm in thickness were cut from the starting material. The disc-shaped samples were HPT-processed by 1/4 (90°), 1/2 (180°), 1 (360°) or 2 (720°) rotations at a speed of 0.2 rpm under a pressure of 7.5 GPa at ambient temperature. The shear strain $\gamma$ of the HPT-processed samples is given by the following formula [3]

$$\gamma = \frac{2\pi NR}{h}$$

where, $N$ is the number of rotation, $R$ is the radial distance from the center and $h$ is the thickness of the sample. After HPT processing, the 2-rotation HPT-processed samples were...
subsequently heat-treated in a salt bath at temperatures ranging from 300 °C to 650 °C for 600 s. The microstructures on transverse sections at a distance of 3.0 mm from the center of the samples were observed by transmission electron microscopy (TEM, JEOL JEM2010) operated at 200 kV and by a field emission scanning electron microscope (FE-SEM, FEI XL30S-FEG) equipped with an electron backscattering diffraction (EBSD) system. The samples for TEM and EBSD observations were electro-polished in an electrolyte (90 vol.% ethanol + 10 vol.% perchloric acid). The mechanical property of the HPT-processed and heat-treated samples was investigated by tensile tests at an initial strain rate of 8.3×10^{-4} s^{-1} at room temperature. The samples for tensile tests were cut from the discs, which had a gauge length of 2.0 mm, width of 1.0 mm and thickness of 0.5 mm, as illustrated schematically in Ref. [9]. The gauge center had a distance of 3.0 mm from the center of the discs and corresponded to the TEM and EBSD observation areas.

3. Results and discussion

3.1. Microstructure evolution during HPT processing

![Figure 1](tem_images.png)

**Figure 1** TEM bright-field images of the HPT-processed samples with different shear strains: (a) 5.55; (b) 11.1; (c) 22.2; (d) 44.4, corresponding to 1/4 (90°), 1/2 (180°), 1 (360°) and 2 (720°) rotations.

The as-received material had a fully annealed austenite (FCC) structure with mean grain size of 35 μm. The fractions of high-angle grain boundaries (HAGB) and twin boundaries are 0.95 and 0.33, respectively [8]. During the HPT processing, the material was refined and martensitic transformation was also induced during the deformation. After HPT processing with a shear strain of 5.55, microstructure consists of lamellar structure, as shown in **Figure 1(a)**. The lamellae are elongated along the shear direction and have a spacing of around 50 nm. When the shear strain increased to 11.1, the lamellae spacing decreased to about 20 nm (**Figure 1(b)**). With further increase in shear strain, the lamellar boundaries became ambiguous in **Figure 1(c, d)**. Even after HPT processing with a much higher shear strain of 222, the material still had a lamellar structure without significant change from a shear strain of 22.2 (360°). [8].
Figure 2 shows a selected area electron diffraction (SAED) pattern of the HPT-processed material with a shear strain of 44.4. The SAED pattern indicates the coexistence of FCC and BCC phases, suggesting that the HPT-processed material has a mixed structure of retained austenite and deformation-induced martensite. The fraction of the deformation-induced martensite was 53% according to the measurement by X-ray diffraction. The deformation-induced martensite and retained austenite were concurrently deformed during the HPT processing. Such a situation is significantly different from other single-phase materials without phase transformation during deformation. The structure of single-phase materials used to saturate once HPT processing reached a critical shear strain. After saturation, the microstructure including the grain size and structure morphology didn't change even after processing with a much large strain. The minimum grain size of the saturated structure was related to the kind of materials [10, 11]. In this study, the lamellar structure and two phases would be favorable to form newly formed austenite having mean grain sizes smaller than 1 μm during the subsequent heat treatment.

3.2. Microstructure evolution during subsequent heat treatment

Figure 3  TEM images of the samples HPT-processed with a shear strain of 44.4 and
subsequently heat treated at different temperatures for 600 s: (a) 300 °C; (b) 500 °C; (c) 550 °C; (d) 650 °C.

The HPT-processed specimens were composed of deformation-induced martensite and retained austenite. During subsequent heat treatment, the retained austenite underwent recovery at low temperatures and recovery/recrystallization at high temperatures. The reverse transformation from deformation-induced martensite to new austenite was also induced above martensite-to-austenite starting temperature. Figure 3 shows the microstructures of the HPT-processed material after heat treatment at different temperatures. The material still has a lamellar structure with a spacing of about 20 nm after the heat treatment at 300 °C (Figure 3a). On the other hand, the lamellar structure vanished and equiaxed grains formed after heat treatment at 500 °C (Figure 3b). The fraction of martensite measured by EBSD was 5%. This suggested that the reverse transformation was not accomplished during the heat treatment at 500 °C. The newly formed austenite had an average grain size of 0.32 μm, which was measured by a linear interception method on the EBSD boundary map.

The microstructure after heat treatments at 530 °C consisted of fully annealed austenite structure, indicating that the reverse transformation of the deformation-induced martensite and recovery/recrystallization of the retained austenite were completed within 600 s [8]. With the increase in the heat treatment temperature to 550 °C and 650 °C, the kinetics of reverse transformation and recovery/recrystallization was accelerated and grain growth of the newly formed austenite followed the two processes. The fully annealed austenite after heat treatment at 550 °C and 650 °C had average grain size of 0.62 μm (Figure 3c) and 1.1 μm (Figure 3d), and the HAGB fractions were 0.91 and 0.92, respectively.

The UFG and NC austenite was formed by the concurrent reverse transformation from deformation-induced martensite to austenite and recovery/recrystallization of highly deformed austenite. The HPT processing introduced a large amount of crystalline defects which could act as nucleation sites for the reverse transformation and recrystallization. It was reported that HPT processing and following heat treatment had been also applied to single-phase materials without transformation [12, 13]. However, the grain sizes obtained in the single-phase materials rapidly increased to several microns after heat treatments. In contrast, the grain size of the newly formed austenite didn’t increased so much even if the heat treatment temperature increased from 550 °C to 650 °C. The present HPT-processed material had two phases, i.e., austenite and martensite. The martensite (the second phase) before completing reverse transformation could inhibit the grain growth of the newly formed or recrystallized austenite. This is also favorable to the formation of the UFG and NC austenite with equiaxed grains.

3.3. Tensile behavior of UFG and NC austenite
Figure 4 shows the tensile stress-strain curves of the starting material, HPT-processed material and heat-treated materials. The coarse-grained austenite (starting material) showed a yield strength of 145 MPa and a total elongation of 160%. Serrations on the stress-strain curve suggest the occurrence of deformation-induced martensite transformation which also contributes to the large ductility. The HPT-processed material showed a significantly enhanced yield strength as high as 2400 MPa but a quite limited uniform elongation. After the heat treatments, the yield strength greatly decreased below 1000 MPa with the increase in the austenitic grain size, but the ductility increased above 65% at the same time, especially for the fully annealed austenite obtained at 550 °C. This distinguished ductility is much larger than that of previously reported NC austenite [14-16]. Additionally, serrations could be still observed on the stress-strain curves of the UFG and NC austenite. This indicates that the transformation-induced plasticity also works in the UFG and NC austenite, similar to the coarse-grained austenite. It should be emphasized that the transformation-induced plasticity of metastable austenite strongly depends on its thermal stability which is affected by the test temperature and the martensite start (Ms) temperature. The Ms temperature could be greatly decreased by grain refinement [17]. The grain refinement was achieved in this study by HPT processing and subsequent heat treatment. However, the mechanical property of the metastable austenite shown in Figure 4 was only investigated at ambient temperature, which has different meanings to the austenite with different grain sizes. The effect of test temperature on the tensile behavior of the UFG and NC metastable austenite would be systematically studied in the future.

4. Conclusions
Fully annealed UFG and NC austenite in a Fe-24Ni-0.3C (wt.%) alloy was prepared by HPT processing and subsequent heat treatment in this study. The HPT-processed specimens showed the lamellar structures with a lamellae spacing below 50 nm, which constituted of deformation-induced martensite and retained austenite. The concurrent reverse transformation of the deformation-induced martensite into austenite and recovery/recrystallization of the highly deformed austenite in the HPT-processed materials were completed at the temperature above 500 °C within 600 s. These two different
processes (reverse transformation and recrystallization) resulted in the UFG and NC austenite with fully annealed structures. The UFG and NC austenite exhibited high yield strength and large ductility which was attributed to the deformation-induced martensitic transformation during the tensile tests.

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