

# HIGH STRENGTH AND GOOD DUCTILITY OF BULK NANOSTRUCTURED META-STABLE AUSTENITIC ALLOYS

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## ABSTRACT

The structural nano-materials have been investigated all over the world in recent years. Severe deformation techniques have been applied to different materials such as Al, Cu, Ti and steels. Such techniques greatly reduced the grain size and improved the yield and tensile strengths. However, the elongation of the materials greatly decreased due to the small amount of work hardening. Therefore, this study has been carried out as a fundamental research for developing austenitic steels with high strength and good elongation using conventional rolling and annealing processes. Fe-0.1%C-10%Cr-5%Ni-8%Mn alloy was melted, homogenized, hot rolled, and cold rolled at room temperature to transform  $\gamma$  austenite to  $\alpha'$  martensite. After that, the specimens were annealed just above its reverse transformation finish temperature ( $A_f$ ) to obtain the fine reversed austenite grains. The grain size of the metastable austenitic steel was successfully refined to less than 200nm by repeating rolling and annealing processes. The resultant nanocrystalline material shows not only high strength but also large elongation because the work hardening ability is enhanced by the strain-induced martensitic transformation during the tensile test.

## KEYWORDS

High Strength, Ductility, Metastable Austenite, Reverse Transformation, Martensite, Grain Refining

## INTRODUCTION

The mechanical properties of nanocrystalline materials are expected to have high strength and good low temperature toughness according to the Hall-Petch relationship as well as the excellent superplasticity at relatively high strain rates and/or low temperatures [1-4]. The severe plastic deformation techniques, such as high pressure torsion (HPT), equal channel angular pressing (ECAP), and accumulative roll-bonding (ARB) [2,6,7], have successfully refined the coarse grains of the materials to the grain sizes ranging from a few ten to a few hundred nanometers. But, the first two methods, i.e. HPT and ECAP exhibit great limits on the materials dimensions. The nanocrystalline samples fabricated by HPT are typically disk-shape of 10 to 20 mm in diameter and 0.2 to 0.5 mm in thickness, and the specimen size of the materials produced by the ECAP are usually less than  $20 \times 20 \times 100 \text{ mm}^3$  [2,5]. These two methods are not suitable for practical applications, especially for large-sized structural materials.

Though ARB shows some advantages in producing large scaled sheets, it seems to still have some problems like surface contamination of the sheets, and the grain size generated by ARB are usually around 500nm [6]. The nanocrystalline materials made by these severe plastic deformation techniques show heavily deformed microstructures and have non-equilibrium grain boundaries with excess interfacial energy and long-range elastic stresses [8-10]. Dynamic recovery can easily be facilitated in this kind of microstructure with high internal energy, intensifying the low work hardening ability, which has already been deteriorated due to the low dislocation storage efficiency

inside the nano-sized grains [11-13]. Thus the tensile elongation to failure of these nanocrystallines rarely exceeds 10%, even for the metals that are very ductile at conventional grain sizes.

Takaki and his co-workers first reported that the reverse transformation from the strain-induced  $\alpha'$  martensite to  $\gamma$  austenite is an effective way to refine the austenite grain size in metastable austenitic stainless steels [14]. This process is characterized by heavy cold rolling to induce the martensitic transformation, followed by annealing for the strain-induced martensite to reversely transform to austenite. They could refine the grain size to 500nm in a 16%Cr-10%Ni steel, and obtain fine grains of 200 to 300nm and a good combination of tensile strength (1127MPa) and total elongation (28%) in a 12.5%Cr-9.5%Ni-2%Mo-0.1%N steel [15].

The objective of this study is to reduce the grain size finer down to less than 200 nm and to develop austenite steels having higher tensile strength and larger elongation by using enhanced thermomechanical processes.

## EXPERIMENTAL PROCEDURES

A Fe-0.1%C-10%Cr-5%Ni-8%Mn alloy was prepared using a high frequency induction furnace in a vacuum atmosphere. The ingot was homogenized at 1100°C for 12 hours under a protective atmosphere, and hot-rolled into the plates of 12mm thick. Two different thermomechanical processes were performed to these plates, as schematically shown in Fig. 1. In the first process (Fig. 1(a)), the cold rolling and annealing were done twice to make the grains finer by repeating the same process. The second process (Fig. 1(b)) was designed to refine the grain size further through the recrystallization of reversed austenite deformed by warm rolling. These two processes will, hereafter, be named as second cycle process (SCP) and recrystallization process (RP), respectively. The reverse transformation start and finish temperatures ( $A_s$  and  $A_f$ ) were determined from dilatational curves measured during the heat-up of the cold-rolled sheets, and the annealing for the reverse transformation was carried out using a salt bath to heat the specimens up to the  $A_f+10^\circ\text{C}$  and hold there for 10 min.

The microstructures were observed using a transmission electron microscope (TEM). Thin foils for TEM investigation were jet-polished in a solution of 10% perchloric acid + 90% ethanol at  $-40^\circ\text{C}$ , and observed in a JEM 2000 EX operating at 160KV. The phase composition was identified by using X-ray diffraction with Cu  $K\alpha$  radiation.

Tensile tests were performed using Instron 1127 machine at the crosshead speed of 2mm/min at room temperature. The size of the gauge part of the tensile specimen for SCP and RP processes was 6mm wide, 1.1mm thick, and 15mm long.

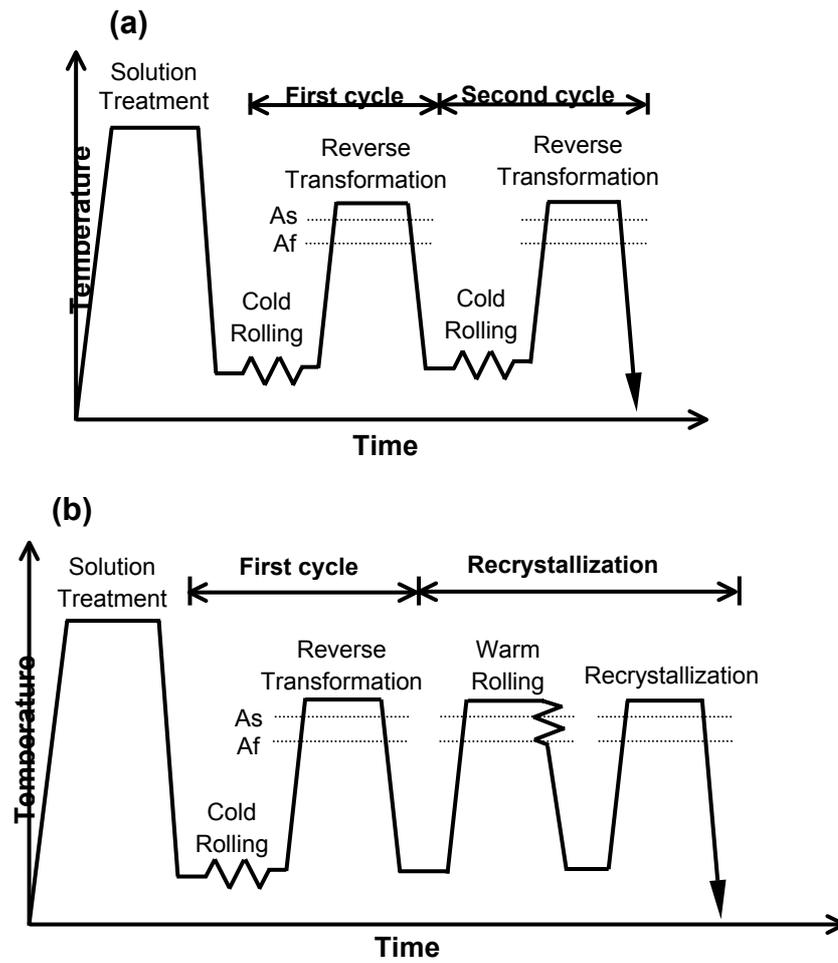


Fig. 1 Schematic illustrations of the two different thermomechanical processes (a) second cycle process (SCP), and (b) recrystallization process (RP)

## RESULTS AND DISCUSSION

### 1. MICROSTRUCTURE

The microstructural changes investigated by X-ray diffraction at each stage of SCP and RP processes were shown in Fig. 2 and Fig. 3, respectively. The microstructure of the solution-treated specimen consists of a mixture of  $\gamma$ -austenite (fcc),  $\alpha'$ -martensite (bcc), and  $\epsilon$ -martensite (hcp), respectively. This means that the martensitic transformation start temperature ( $M_s$ ) of the solution-treated specimen is higher than the room temperature.

After the first cold rolling, as shown by the X-ray pattern (2) in Fig. 2, the material is completely  $\alpha'$ -martensite, which is induced by heavy cold rolling. After the first reverse transformation, the  $\alpha'$ -martensite reversely transformed to  $\gamma$ -austenite, and no peaks of martensite could be monitored from the pattern (3) of Fig. 2. This indicates that  $M_s$  temperature has dropped to below room temperature after reverse transformation annealing due to the refinement of grain size.

In order to reduce the grain size further, additional thermomechanical processes (SCP and RP) were taken, as shown in Fig. 1. For SCP process, after the second cold rolling ( X-ray pattern (4) in Fig. 2 ), the sample is mostly  $\alpha'$ -martensite with little amount of retained  $\gamma$ -austenite. For RP process, after the warm rolling the peaks of  $\gamma$ -austenite remain intense, indicating that the microstructure of the warm-rolled specimen is mainly deformed  $\gamma$ -austenite, as was expected. After the second annealing, both specimens of SCP and RP processes reveal single  $\gamma$ -austenite phase. However, there are some differences in the intensities of several  $\gamma$ -austenite peaks, probably due to the different textures depending on the thermomechanical processes. The X-ray diffraction patterns of both cold and warm rolled specimens show obviously peak broadening due to the strong internal stress.

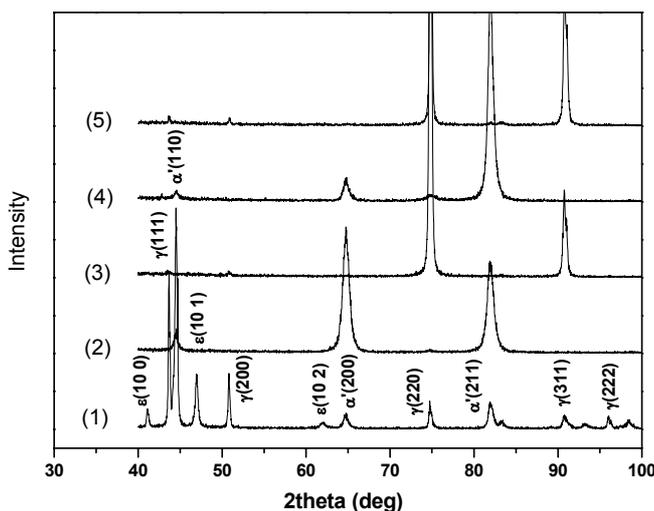


Fig. 2 Microstructural changes during SCP process

- (1) after solution treatment,
- (2) after the first cold rolling,
- (3) after the first reverse transformation,
- (4) after the second cold rolling, and
- (5) after the second reverse transformation

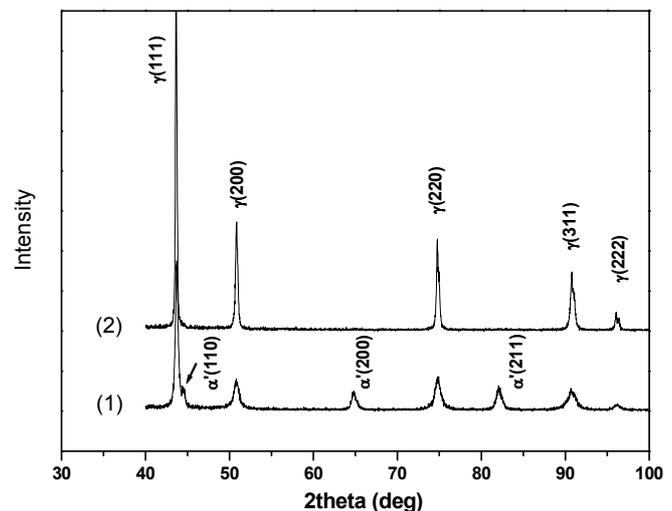


Fig. 3 Microstructural changes during RP process

- (1) after warm rolling, and
- (2) after recrystallization

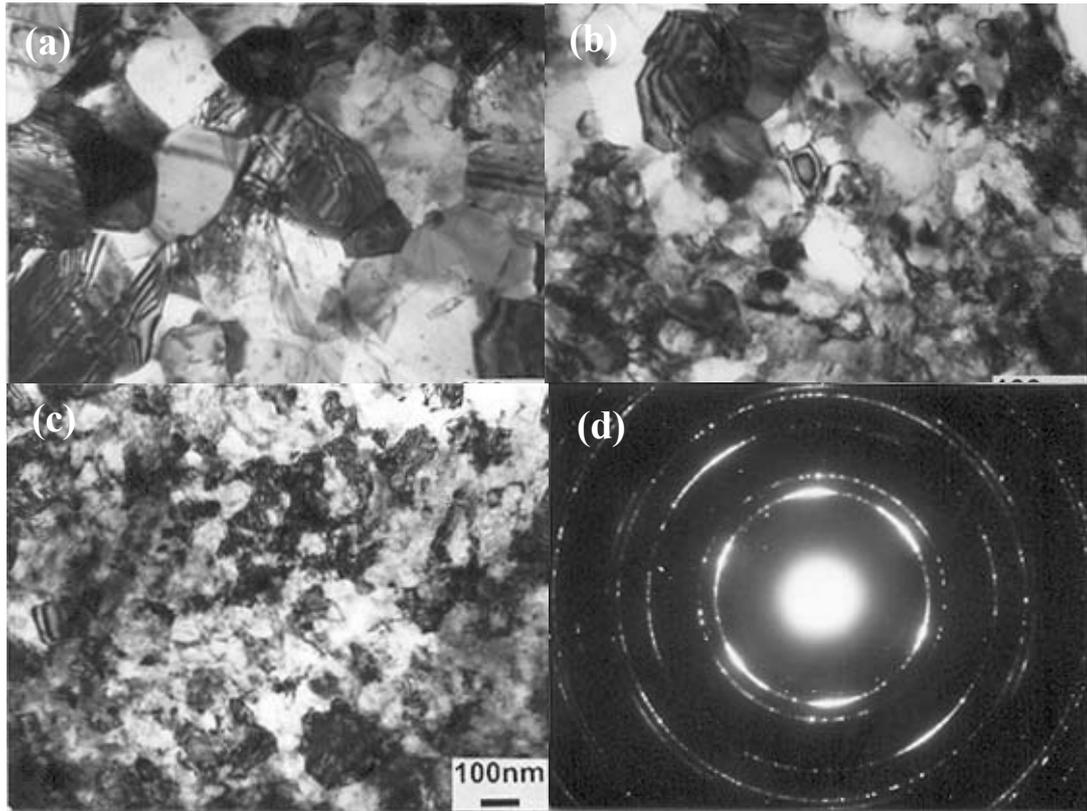


Fig. 4 TEM micrographs showing nanocrystallines made from different treatments (a) after the first reverse transformation, (b) after SCP process, (c) after RP process, and (d) SAED pattern of (b)

Figure 4 shows the TEM microstructural changes depending on the thermomechanical processes. After the first cycle, the microstructure is relatively homogeneous, showing an average grain size of 300nm and distinct grain boundaries. This microstructure is clearly different from those fabricated by other severe plastic deformation techniques. For example, the grain boundaries in nanocrystallines made by HPT and ECAP are generally curved or wavy, and poorly defined [2,8]. But in case of this process, new  $\gamma$ -austenite grains nucleate and gradually grow up from the strain-induced  $\alpha'$ -martensite during the reverse transformation. The residual internal stress in the reverse austenite is probably less than that in the specimens fabricated by other severe plastic deformation techniques.

After the SCP process, the grain size of the specimen is successfully reduced to less than 200nm, as shown in the Fig. 4 (b). But the homogeneity of the grain sizes seems to be decreased compared with the grains after the first thermomechanical treatment (Fig. 4(a)). Some grains are even smaller than 100nm, and others are a little larger than 200nm. Although subgrains may exist in the morphology, the relative uniformity of the diffraction rings in the SAED pattern (aperture area approximately  $0.75\mu\text{m}^2$ ) reveals that most of the neighboring grains have high-angle boundaries.

After the RP process, as shown in Fig. 4(c), the grain size is around 100nm. The selected area diffraction also revealed almost the similar pattern as Fig. 4 (d), which can be indexed by  $\gamma$ -austenite. But the grain boundaries are not very clear, and some of them are difficult to distinguish, just like the microstructures observed in HPT or ECAP samples.

Since the repetitive thermomechanical treatment is easy to achieve by the conventional cold rolling and annealing, these processes (SCP and RP) seem suitable for large sized sheets with nanocrystalline microstructure, as compared with other severe plastic deformation techniques.

## 2. MECHANICAL PROPERTIES

Figure 5 presents the tensile stress-strain curves of the specimens after different treatments. As-hot rolled state is also given for comparison, and their mechanical properties were listed in Table 2. Hot rolled specimen shows low yield strength and large elongation. After the first reverse transformation, the yield strength is greatly increased to 708MPa, and the tensile strength is 1070MPa. The elongation is also good, which is 36.0%. After the SCP process, the yield and the tensile strengths increased further to 779 and 1102MPa, respectively. But the elongation shows some decrease (32.0%). After the RP process, the specimen shows the best performance in this study with the highest yield and tensile strengths (915 and 1146MPa, respectively) as well as the highest elongation (42.5%).

Table 2 Tensile properties after different treatments

Thermomechanical process	Yield strength (MPa)	Tensile strength (MPa)	Elongation (%)
as-hot rolled	120	1041	38.9
after the first reverse transformation	708	1070	36.0
after SCP process	779	1102	32.0
after RP process	915	1146	42.5

Previous researches revealed that it is normal for nanocrystalline materials to show much higher strength than their coarse-grained counterparts, as expected from the well-known Hall-Petch relationship. But, the elongation of the most nanocrystalline materials is dramatically decreased, due to the weak work hardening ability, which greatly reduces the uniform elongation prior to necking under uniaxial tension [12,13]. But, for the Fe-0.1%C-10%Cr-5%Ni-8%Mn alloy studied in this paper, the work hardening ability of its nanocrystallines is enhanced by the strain-induced martensitic transformation during tensile tests, so as to exhibit long uniform strain and good elongation.

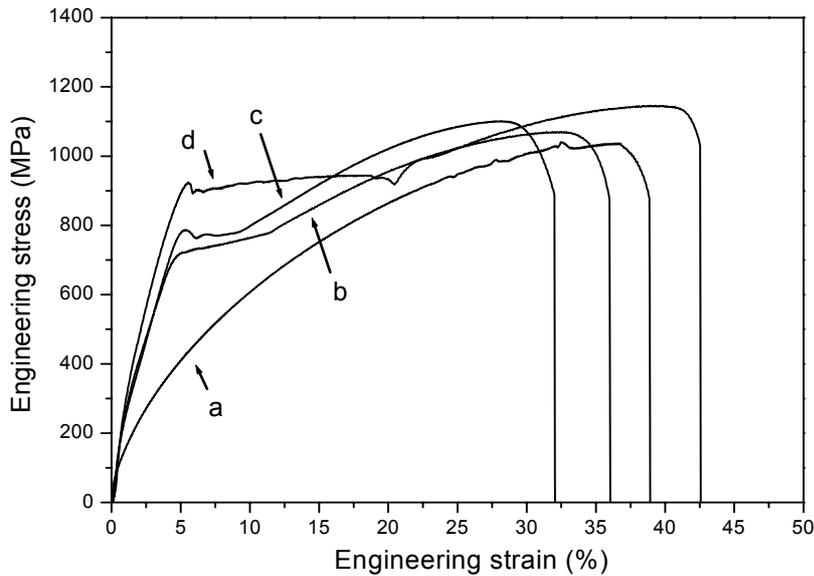


Fig. 5 Tensile stress-strain curves after different treatments  
 (a) as-hot rolled, (b) after first reverse transformation,  
 (c) after SCP process, and (d) after RP process

## CONCLUSIONS

The grain size of a metastable austenitic steel Fe-0.1%C-10%Cr-5%Ni-8%Mn was refined to less than 200 nm using phase transformations occurring during the conventional rolling and annealing process. The nanocrystalline steel shows a good combination of high strength and good elongation. This technique shows great advantages for practical application, especially for large-sized sheets, as compared with the other severe plastic deformation techniques.

## REFERENCES

- 1) H. GLEITER, *Progr. Mater. Sci.*, **33** (1991), p.223.
- 2) R.Z. VALIEV, R.K. ISLAMGALIEV, and I.V. ALEXANDROV, *Progr. Mater. Sci.*, **45**, (2000), p.103.
- 3) H. GLEITER, *Acta Mater.*, **48**, (2000), p.1.
- 4) J.R. WEERTMAN, *Mater. Sci. Eng. A*, **166**, (1993), p.161.
- 5) R. Z. VALIEV, *Met. Mater.-Int.*, **7**, (2001), p.413.
- 6) Y. SAITO, N. TSUJI, H. UTSUNOMIYA, T. SAKAI, and R.G. HONG, *Scripta Mater.*, **39**, (1998), p.1221.
- 7) Y.T. ZHU, H. G. JIANG, J.Y. HUANG, and T.C. LOWE, *Metall. Mater. Trans.*, **32A**, (2001), p.1559.
- 8) V.N. CHUVIL'DEEV, V.I. KOPYLOV, and W. ZEIGER, *Ann. Chim. Sci. Mat.*, **27**, (2002), p.55.

- 9) R.K. ISLAMGALIEV, and R.Z. VALIEV, Fiz. Met. Metalloved, 87, (1999), p.46.
- 10) R. Z. VALIEV, V. Yu GERTSMAN, and O.A. KAIBYSHEV, Phys. Sta. Sol. (a), 97, (1986), p.11.
- 11) J. SCHIØTZ, F.D. DITOLLA, and K.W. JACOBSEN, Nature, 391, (1998), p.561.
- 12) D. JIA, Y.M. WANG, K.T. RAMESH, and E. MA etc, Appl. Phys. Letter, 79, (2001), p.611.
- 13) R.Z. VALIEV, Mater. Sci. Eng., A234-236, (1997), p.59.
- 14) K. TOMIMURA, S. TAKAKI, and Y. TOKUNAGA, ISIJ Inter., 31, (1991), p.1431.
- 15) S. TAKAKI, ISIJ, Japan, 74, (1988), p.1052.

### **IMPORTANT NOTE**

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