Met. Mater. Int., Vol. 17, No. 4 (2011), pp. 553~556
doi: 10.1007/s12540-011-0804-x Published 31 August 2011

Effect of Grain Size on Strain-Induced Martensitic Transformation Start Temperature in an Ultrafine Grained Metastable Austenitic Steel

Yeon-Seung Jung[1], Young-Kook Lee[1,*], David K. Matlock[2], and Martin C. Mataya[2]

[1]Department of Materials Science and Engineering, Yonsei University, Seoul 120-749, Korea
[2]Advanced Steel Processing and Products Research Center, Colorado School of Mines, Golden, CO 80401, USA

(received date: 25 November 2010 / accepted date: 18 February 2011)

Tensile tests were used to investigate the effect of grain size on the strain-induced martensitic transformation start temperature in metastable austenitic steel with special attention to ultrafine grain size. The austenite grains were refined to submicron size by the strain-induced martensite and its reverse transformations (SIMRT), which occurred during a conventional cold rolling and annealing process. The start temperature of the strain-induced martensitic transformation was linearly lowered with a decrease in austenite grain size, even down to submicron grain sizes. This result is due to the decrease in grain size causing an increase in the temperature dependency of the strain-induced martensitic transformation and higher austenite stability brought about by grain refinement.

Keywords: nanostructured materials, thermomechanical processing, phase transformation, tensile test, grain size

1. INTRODUCTION

Metastable austenite, which can transform to martensite via mechanical work [1,2], is present in many types of austenitic stainless steel (including Types 301 and 304) and TRansformation-Induced Plasticity (TRIP) steel. The phase transformation of metastable austenite to martensite with plastic strain occurs between M_s^σ, a critical temperature above which martensite forms after the onset of plastic flow in the austenite, and M_d, a critical temperature above which austenite is stable and does not transform to martensite by deformation [2]. Strain-induced martensitic transformation increases the strain hardening rate, leading to excellent combinations of strength and elongation, as displayed at room temperature in metastable austenitic stainless steels [3-7] and some TRIP steels [8,9].

The transformation of metastable austenite depends on multiple parameters, including grain size [5,9], strain rate [3,4,9], and alloy addition [4,5]. Of these parameters, austenite grain size is extremely important, since a decrease in grain size improves austenite stability, retards strain-induced martensitic transformation, and lowers the M_d temperature [5,9]. However, studies on grain size effects have been limited to grain sizes typical of conventional processing routes as there is limited reported information on variation in M_d temperature in metastable austenite with submicron-sized ultrafine grains (UFG).

Accordingly, the objective of the present study is to investigate the effect of grain size on the M_d temperature of UFG metastable austenitic steel and to suggest a new M_d temperature equation that is applicable to materials with submicron austenitic grain sizes.

2. EXPERIMENTAL PROCEDURE

A metastable austenitic steel, with a composition of Fe-0.01%C-8.05%Ni-9.85%Cr-7.80%Mn in weight percent, was vacuum-melted, cast into an ingot, homogenized at 1200 °C for 12 h under a protective nitrogen gas atmosphere, and finally hot-rolled at 1100 °C into a 12 mm thick plate. The microstructure of the hot-rolled plate was fully austenite, as confirmed by X-ray diffraction (XRD). Cold rolling was performed with a thickness reduction of about 80% to transform the metastable austenite to martensite. The fully martensite structure in the cold-rolled sheet was also confirmed by XRD. The cold-rolled specimens were annealed for 5 min at 593 °C, 616 °C, 646 °C, 693 °C, and 804 °C using a salt bath to form reversed austenite with different grain sizes. This Strain-Induced Martensite and its Reverse Transformation (SIMRT) process can easily fabricate submicron-sized UFG microstructures in metastable

*Corresponding author: yklee@yonsei.ac.kr
©KIM and Springer

austenitic steels [10,11].

Annealing temperatures were determined based on the reverse transformation start (A_s) and finish (A_f) temperatures, which were measured from a dilatational curve obtained while the cold-rolled specimen was heated at a rate of 10 °C s^{-1}. In addition, to obtain larger grain sizes (12 μm), one specimen was annealed at 900 °C for 30 min in a box furnace.

The microstructures of the annealed specimens were observed for grain size measurement using a transmission electron microscope operating at 160 kV. Thin TEM foils were jet-polished at 15 °C in a solution of 10 % perchloric acid and 90 % acetic acid. Metallographic samples for analysis with both light and scanning electron microscopy techniques were mechanically and electrochemically polished using a solution of 10 % perchloric acid and 90 % acetic acid at 20 V. Subsequently, the specimens were etched with a mixed solution of 3.5 % hydrochloric acid, 6.5 % distilled water, and 90 % ethyl alcohol to reveal the austenite grain boundaries. The grain sizes of the annealed specimens (single phase austenite) were measured by applying the linear intercept method [12] to the micrographs. Specimens annealed at 593 °C and 616 °C had ultrafine grains with average sizes of 0.25 μm and 0.55 μm, respectively. Grain size (>2 μm) of the remainder of the samples increased with annealing temperature to a maximum of 12 μm for the sample annealed for 30 min at 900 °C.

Isothermal tensile tests were performed with a commercial floor model electro-mechanical test machine equipped with a special fixture for testing in an isothermal oil bath between room temperature (22 °C) and 140 °C. All samples were submerged in the heating fluid and tested at a constant crosshead speed of 0.1 mm·min^{-1}. The tensile tests at temperatures higher than 140 °C were performed using a commercial test frame equipped with a furnace. Sub-sized tensile specimens with a reduced gage section of 25.0 mm long × 6.35 mm wide × 2.21 mm thick were used.

A two-step process was used to investigate the temperature dependency of the formation of strain-induced martensite and the effect of grain size on M_d temperature. First, tensile specimens were deformed to the point of fracture at different isothermal temperatures. Then, the martensite volume fractions (MVF) were measured near the fracture within the neck of the specimens by using Cu-Kα radiation to obtain X-ray diffracted peak intensities [13]. The scanning speed and range (2θ) were 2° min^{-1} and between 40° and 100°, respectively.

3. RESULTS AND DISCUSSION

Figure 1 shows the plots of MVF vs. tensile test temperature for each grain size. In all specimens, when the test temperature increased, the volume fraction of strain-induced martensite decreased because elevated temperatures reduce the chemical driving force for martensitic transformation. Furthermore, Cohen and Olson [14] reported that the strain-induced martensitic transformation kinetics is delayed with increasing deformation temperature by using an equation

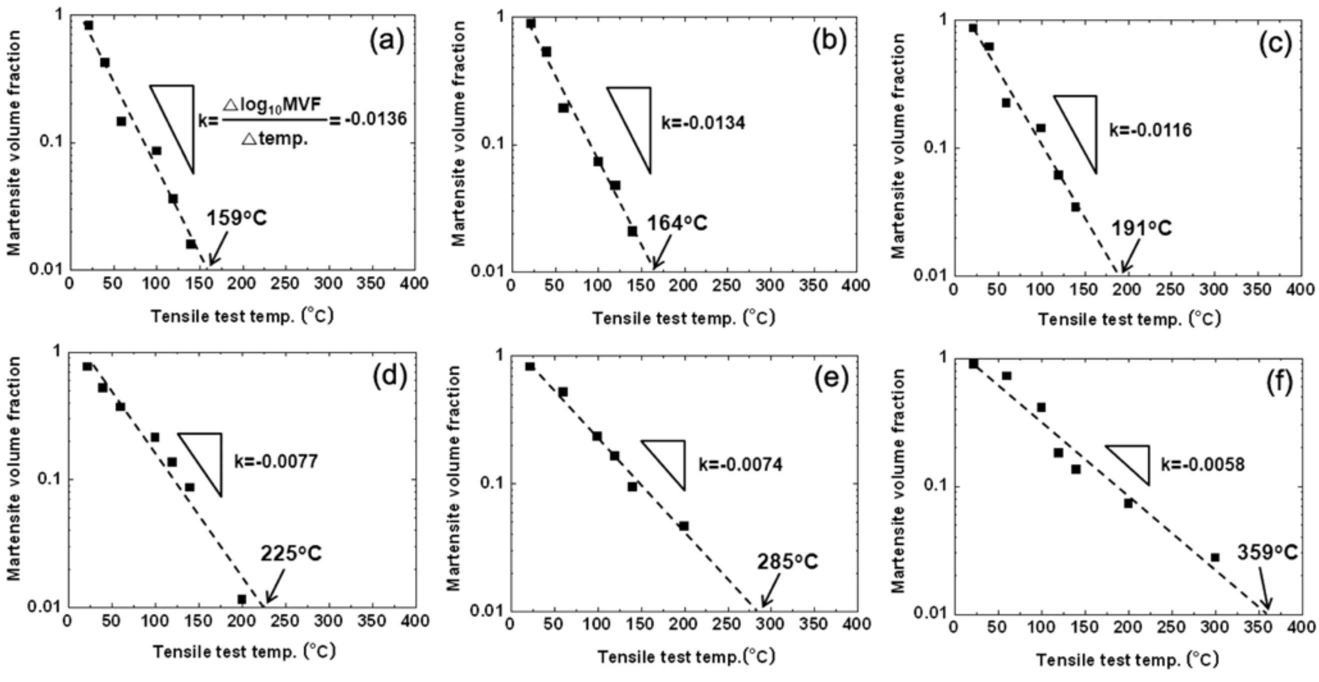

Fig. 1. The plots of martensite volume fraction vs. tensile test temperature for each average grain size: (a) 0.25 μm, (b) 0.55 μm, (c) 1.8 μm, (d) 3.8 μm, (e) 7.2 μm, and (f) 12 μm. The martensite volume fraction was measured by XRD tests at the fractured edges of tensile specimens.

of the form:

$$f^{\alpha'} = 1 - \exp\{-\beta[1 - \exp(-\alpha\varepsilon)]^n\} \quad (1)$$

where $f^{\alpha'}$ is the volume fraction of α' martensite, ε is plastic strain, n is a constant relating to the number of shear-band intersections with plastic strain. In this equation, α indicates the rate of shear-band formation, which is inversely dependent on both stacking fault energy and deformation temperature. The shear-bands are ε martensite, mechanical twins, and stacking-fault bundles. Here, β represents the probability of embryo formation of α' martensite at shear-band intersections, and this value also decreases above a critical deformation temperature.

Therefore, negative slopes in the plots of MVF vs. tensile test temperature (Fig. 1) are thought to be attributed to the reduction in both chemical driving force and the kinetics of the strain-induced martensitic transformation caused by the increase in deformation temperature.

Meanwhile, the magnitude of the slope in Fig. 1 decreased with an increase in grain size. As shown in Figs. 1(a) and (b), specifically the 0.25 μm and 0.55 μm specimens had steep slopes, indicative of strong temperature dependence of the strain-induced martensitic transformation. On the other hand, the coarse-grained (CG) specimen with the grain size of 12 μm (Fig. 1(f)) had a gentle slope, indicating relatively weak temperature dependency. Because grain refinement increases SFE and austenite stability [15], both α and β values in Eq. 1 for the UFG specimens should be smaller than those for the CG specimen.

In each figure, the data, which are interpreted as a linear function, were extrapolated to determine the temperature corresponding to a MVF of 0.01, assumed here to be essentially zero. The intercept temperature was taken as the M_d temperature, which is plotted as a function of grain size in Fig. 2. Although the M_d temperatures may be a bit scattered

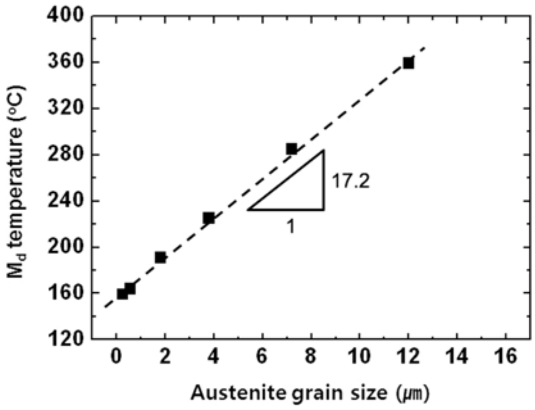

Fig. 2. Change of M_d temperature with austenite grain size in Fe-0.01%C-8.05%Ni-9.85%Cr-7.80%Mn steel.

because of the limited accuracy of the phase volume fractions measured by XRD analysis [13], there is an obvious linear relationship between austenite grain size (d) and M_d temperature in the specimens with an average grain size of less than 12 μm. This linear relationship is effective even for submicron grain sizes.

$$M_d(^\circ C) = 17.2 \times d\ (\mu m) + 158 \quad (2)$$

However, it is not certain that this linear relationship is applicable to specimens with coarse grains much larger than 12 μm. For metastable austenitic stainless steels (Types 301 and 304) with relatively coarse grains, the M_{d30} temperature, above which austenite does not transform to martensite in the specimen strained by 0.3, increased logarithmically with grain size, as shown in Fig. 3 [5]. Thus, the M_{d30} temperature rose only by approximately 10 °C as the grain size increased from 10 μm to 100 μm. However, both stainless steels exhibited a linear relationship between M_d temperature and grain size when the grain size was between 7 μm and 20 μm, which is similar to our results shown in Fig. 2.

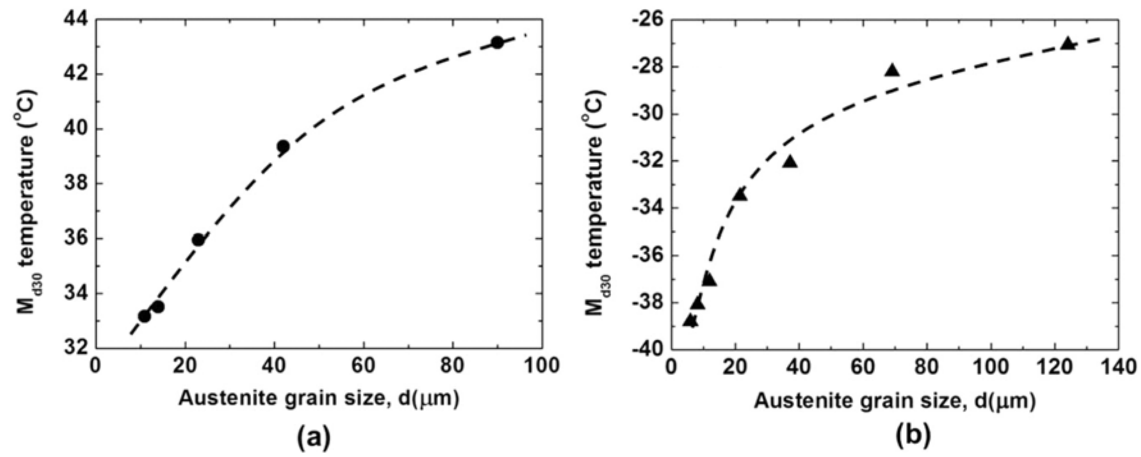

Fig. 3. Changes of M_{d30} temperature with austenite grain size in (a) 301 stainless steel and (b) 304 stainless steel [5].

5. CONCLUSIONS

The strain-induced martensite start (M_d) temperature was investigated by tensile tests using specimens with various grain sizes, with particular focus on submicron grain sizes, which were fabricated by applying the strain-induced martensite and its reverse transformations (SIMRT) process to metastable austenitic steel. The M_d temperature was linearly decreased when the grain size was reduced from 12 μm to the ultrafine grain size of 0.25 μm.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the support of the sponsors of the Advanced Steel Processing and Products Research Center, an industry/university cooperative research center at the Colorado School of Mines and the Fundamental R&D Program for Core Technology of Materials funded by the Ministry of Commerce, Industry and Energy, Republic of Korea.

REFERENCES

1. G. B. Olson and M. Cohen, *Metall. Trans. A* **3A**, 1907 (1982).
2. D. K. Matlock, G. Krauss, and F. Zia-Ebrahimi, *ASM Materials Science Seminar* (ed., G. Krauss), p. 47, ASM, St. Louis, USA (1982).
3. S. F. Peterson, M. C. Mataya, and D. K. Matlock, *J. Metals* **49**, 54 (1997).
4. G. L. Huang, D. K. Matlock, and G. Krauss, *Metall. Trans. A* **20A**, 1237 (1989).
5. K. Nohara, Y. Ono, and N. Ohashi, *J. Iron Steel Inst. Jpn.* **63**, 772 (1977).
6. I. Choi, D. Son, S.-J. Kim, D. M. Bruce, and D. K. Matlock, and J. G. Speer, *Met. Mater. Int.* **12**, 13 (2006).
7. D. W. Kim, C. H. Han, and B. S. Lee, *Kor. Inst. Met. & Mater.* **47**, 391 (2009).
8. S.-J. Kim, C. G. Lee, and T.-H. Lee, *Scripta mater.* **48**, 539 (2003).
9. H. Mirzadeh and A. Najafizadeh, *Mater. Character.* **59**, 1650 (2008).
10. Y. Q. Ma, J.-E. Jin, and Y.-K. Lee, *Scripta mater.* **52**, 1311 (2005).
11. Y.-K. Lee, J.-E. Jin, and Y.Q. Ma, *Scripta mater.* **57**, 707 (2007).
12. G. E. Dieter, *Mechanical Metallurgy, 3rd ed.*, p. 78-80, McGraw-Hill, New York (1986).
13. B. D. Cullity and S. R. Stock, *Elements of X-Ray Diffraction, 3rd ed.*, p. 351-355, Prentice Hall, Upper Saddle River (2001).
14. M. Cohen and G. B. Olson, *Metall. Trans. A* **6A**, 791 (1975).
15. Y.-K. Lee and C.-S. Choi, *Metall. Trans. A* **31A**, 2735 (2000).