Structural refinement and property optimization in an Fe-23Cr-8.5Ni duplex stainless steel

Xie, L.; Huang, T. L.; Wang, Y. H.; Zhang, L.; Wu, G. L.; Tsuji, N.; Huang, X.

Published in:
I O P Conference Series: Materials Science and Engineering

Link to article, DOI:
10.1088/1757-899X/219/1/012045

Publication date:
2017

Document Version
Publisher's PDF, also known as Version of record

Link back to DTU Orbit

Citation (APA):
Xie, L., Huang, T. L., Wang, Y. H., Zhang, L., Wu, G. L., Tsuji, N., & Huang, X. (2017). Structural refinement and property optimization in an Fe-23Cr-8.5Ni duplex stainless steel. I O P Conference Series: Materials Science and Engineering, 219. https://doi.org/10.1088/1757-899X/219/1/012045

General rights
Copyright and moral rights for the publications made accessible in the public portal are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

- Users may download and print one copy of any publication from the public portal for the purpose of private study or research.
- You may not further distribute the material or use it for any profit-making activity or commercial gain
- You may freely distribute the URL identifying the publication in the public portal

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.
Structural refinement and property optimization in an Fe-23Cr-8.5Ni duplex stainless steel

To cite this article: L. Xie et al 2017 IOP Conf. Ser.: Mater. Sci. Eng. 219 012045

View the article online for updates and enhancements.

Related content
- Research on damping properties optimization of variable-stiffness plate
  QI Wen-kai, YIN Xian-tao and SHEN Cheng
- Ultrafine grained steels managing both high strength and ductility
  N Tsuji
- Variability of Mechanical Properties and Weight for Reinforcing Bar Produced in Saudi Arabia
  F. Djavanroodi and A. Salman
Structural refinement and property optimization in an Fe-23Cr-8.5Ni duplex stainless steel

L. Xie¹, T.L. Huang¹, Y.H. Wang², L. Zhang¹, G.L. Wu¹,*, N. Tsuji³ and X. Huang⁴

¹College of Materials Science and Engineering, Chongqing University, Chongqing 400045, China
²National Engineering Research Center for Equipment and Technology of Cold Strip Rolling, Yanshan University, Qinhuangdao 066004, China
³Department of Materials Science and Engineering, Kyoto University, Kyoto 606-8501, Japan
⁴Section for Materials Science and Advanced Characterization, Department of Wind Energy, Technical University of Denmark, DK-4000 Roskilde, Denmark

E-mail: wugl@cqu.edu.cn

Abstract. An Fe-23Cr-8.5Ni duplex stainless steel was used to prepare samples with different volume-fraction-weighted grain sizes ($d_{eq}$), ranging from the nano-scale to the micrometer-scale by cold rolling and subsequent annealing. The cold rolled sample with $d_{eq}$ of 72 nm showed a high yield strength of about 1.3 GPa but only a small tensile elongation. An abrupt increase of ductility was observed as $d_{eq}$ increased to 375 nm, resulting in a good combination of yield strength of 738 MPa and tensile elongation of 29%. Further increase of $d_{eq}$ up to the micrometer-scale results in continued decreases in yield strength but with only a limited improvement in the ductility.

1. Introduction

Nanostructured single phase metals produced by large strain deformation usually show a high ultimate tensile strength at a relatively small strain followed by a relatively large post elongation [1]. It is difficult to improve the limited ductility, especially the limited uniform elongation, until the grain size is increased up to the micrometer-scale, as widely observed in Al [2], Cu [3], Ti [4] and IF steel [5].

Several microstructural design principles have been put forward recently to improve the strain hardening ability of nanostructured metals, such as gradient structures [6], heterogeneous lamella structures [7] and dual or multi phases structures [8]. In this context dual-phase nanostructures also show a potential ability to achieve both high strength and good ductility. In the present study, we investigate the structural scale effect on the mechanical properties of a duplex stainless steel (DSS) with structural scales from nanometers to micrometers.
2. Materials and methods
The material used in the present study was an Fe-23Cr-8.5Ni DSS, with chemical composition of 0.001% C, <0.01% Si, <0.01% Mn, <0.001% P, <0.001% S, 8.52% Ni, 22.9% Cr, 0.002% N, 0.04% O, and the balance Fe (mass%). The ingot was homogenized at 1150°C for 2 h and hot forged at 0.001% C, <0.01% Si, <0.01% Mn, <0.001% P, <0.001% S, 8.52% Ni, 22.9% Cr, 0.002% N, 0.04% O, and the balance Fe (mass%). The ingot was homogenized at 1150°C for 2 h and hot forged at temperatures above 900°C. The hot forged plate was then cold rolled to a thickness reduction of 90% and subsequently annealed at temperatures from 700-1000°C for 5 min and 30 min in order to tailor the structure into different scales.

Microstructural characterization was carried out using an Axiovert 40 MAT optical microscope (OM) and a JEOL 2100 transmission electron microscope (TEM). The sampling plane was the longitudinal section containing the rolling direction (RD) and normal direction (ND). Samples for OM characterization were mechanically polished followed by electrochemical polishing in an electrolyte of 15 vol.% perchloric acid and 85 vol.% acetic acid at a voltage of 15 V at room temperature. Thin foils for TEM characterization were prepared by mechanical polishing to a thickness of 70 μm and then electropolishing using a twin-jet polisher in a solution of 25 vol.% perchloric acid and 75 vol.% ethanol at a voltage of 20 V at -20°C. The volume fractions of constituent phases were quantified by X-ray diffraction (XRD) using a Rigaku D/max 2500PC X-ray diffractometer with Cu Kα radiation and a step size of 0.02°. Tensile specimens of gauge dimensions 25 × 5 × 1 mm were machined from the cold rolled and annealed sheets with tensile axis along the RD, and tested at a uniaxial quasi-static strain rate of 6 × 10⁻⁴ s⁻¹.

Structural parameters in the DSS samples include grain size, phase size and volume fractions of the two phases. For simplification, we defined the structural size as the volume-fraction-weighted average grain size $d_{av}$ [9], that is $d_{av} = d_{avf} + d_{ava}$, where $d_{av}$ and $d_{av}$ are the average grain sizes of the ferrite and austenite phases, respectively (determined by an intercept method along the ND) and where $f_{av}$ and $f_{av}$ are the volume factions of the two phases.

3. Results and Discussion

3.1. Microstructural characterization
Figure 1a shows the XRD spectra of the Fe-23Cr-8.5Ni DSS in the hot forged state and after cold rolling. Both ferrite and austenite diffraction peaks can be observed in the hot forged state. The volume factions of the ferrite and austenite phases were determined to be 44.6% and 55.4%, respectively. After 90% cold rolling, the spectrum exhibits an absence of austenite, indicating the occurrence of a deformation-induced martensitic transformation. After annealing of the cold rolled sheet at 700°C or above, the deformation induced martensite (α' martensite) transforms back to austenite. As shown in figure 1b, the austenite reversion is dependent on both the annealing temperature and the holding time. In the case of 5 min annealing, the α' martensite gradually reverts to austenite, resulting in a slow increase of austenite fraction with temperature. The samples annealed for 30 min in the range from 700-1000 °C exhibit a large and almost constant austenite content, close to the amount of austenite in the hot forged state, indicating the completion of austenite reversion.

An example OM image of the 90% cold rolled sample is shown in figure 2a and reveals a microstructure consisting of alternating ferrite and α' martensite lamellae, with dark and bright contrast, respectively. The samples annealed at 700 and 1000 °C for 5 min (figures 2b and 2c) both exhibit a banded structure similar to the cold rolled sample. Upon further annealing for 30 min, as shown in figures 2d and 2e, the microstructures change into a chain-like morphology.

The TEM image of the cold rolled sample shown in Figure 3a reveals a well-defined lamellar structure, with an average boundary spacing of 72 nm. After annealing at 700 °C for 5 min (figure 3b), recovery in ferrite and the reverse transformation from α' martensite to austenite are initiated. The constituent phases in the TEM images were identified by electron diffraction. Some austenite grains are indicated by the white arrows and a diffraction pattern from the circled austenite grain is shown in the insert. The transformed austenite grains have an appearance similar to that of recrystallized austenite grains reported in austenitic stainless steels [11]. As the temperature is increased up to
1000°C, as shown in figure 3c, the austenite reversion continues by consumption of the remaining α′-martensite, and some austenite layers are well formed. The austenite layers consist of equiaxed grains, some of which contain annealing twins. In the ferrite phase, on the other hand, a typical recovered structure is seen. As the annealing time is extended to 30 min, as shown in figures 3d and 3e, further structural coarsening takes place. Table 1 lists the microstructural parameters of all samples investigated in the present experiment. It is seen that DSS samples with structural sizes ranging from 72 nm to 2.1 μm were obtained.

Figure 1. (a) XRD spectra of samples before and after 90% cold rolling. (b) Austenite fraction vs temperature for the cold rolled samples and then annealed at 700-1000°C for 5 and 30 min.

Figure 2. OM images of cold rolled and annealed samples: (a) cold rolled, (b) annealed at 700 °C for 5 min, (c) annealed at 1000 °C for 5 min, (d) annealed at 700 °C for 30 min, (e) annealed at 1000 °C for 30 min.
**Figure 3.** TEM images of cold rolled and annealed samples: (a) cold rolled, (b) annealed at 700°C for 5 min, (c) annealed at 1000°C for 5 min, (d) annealed at 700°C for 30 min, (e) annealed at 1000°C for 30 min.

**Table 1.** Microstructural parameters of cold rolled and annealed samples.

|                  | \(d_{\alpha}\) | \(d_{\gamma}\) | \(f_{\alpha}\) | \(f_{\gamma}\) |
|------------------|----------------|----------------|----------------|----------------|
| CR               | 72 nm          | 72 nm          | 100%           | 0%             |
| CR+700°C×5min    | 169 nm         | 168 nm         | 83.3%          | 16.7%          |
| CR+800°C×5min    | 301 nm         | 294 nm         | 72.2%          | 27.8%          |
| CR+900°C×5min    | 571 nm         | 534 nm         | 71.7%          | 28.3%          |
| CR+1000°C×5min   | 808 nm         | 768 nm         | 67.0%          | 33.0%          |
| CR+700°C×30min   | 375 nm         | 347 nm         | 48.9%          | 51.1%          |
| CR+800°C×30min   | 723 nm         | 621 nm         | 47.6%          | 52.4%          |
| CR+900°C×30min   | 1.1 \(\mu\)m  | 1.1 \(\mu\)m   | 42.8%          | 57.2%          |
| CR+1000°C×30min  | 2.1 \(\mu\)m   | 1.8 \(\mu\)m   | 39.9%          | 60.1%          |

### 3.2. Mechanical properties

Figure 4 shows the engineering stress-strain curves of the 90% cold rolled and annealed samples for volume-fraction-weighted grain sizes \(d_{\alpha\gamma}\) up to 2.1 \(\mu\)m. The cold rolled sample with \(d_{\alpha\gamma}\) of 72 nm has a very high yield strength of about 1.3 GPa, but exhibits an ultimate tensile stress followed by necking at a very early stage of tensile deformation, resulting in a limited uniform elongation of less than 2% and a total elongation below 5%. As the volume-fraction-weighted grain size is increased to \(d_{\alpha\gamma}=169\) nm, the yield strength rapidly decreases to 928 MPa, while the elongation shows almost no
increase. An abrupt increase of ductility is observed as \( d_{\alpha} \) is increased to 375 nm. The yield strength under this condition is 738 MPa with an elongation of 29%. As \( d_{\alpha} \) is further increased the yield strength decreases gradually, but improvements in ductility are limited. A good combination of strength and ductility was therefore achieved in the sample with an average structural size of 375 nm. Under this condition the yield strength is 2.3 times higher than that of the sample with \( d_{\alpha} = 2.1 \) \( \mu \)m, while its tensile elongation (29%) is only a slightly smaller than that (33%) of the latter. Note that samples with \( d_{\alpha} \) ranging from 375 to 808 nm exhibit a discontinuous yielding, characterized by a plateau (1~3% Lüders elongation) in the stress-strain curves. This was also observed in ultrafine grained single phase metals such as Al [2], Cu [3], IF steel [5] and austenitic stainless steels [11]. A continuous yielding is seen for structural sizes above 1 \( \mu \)m.

![Figure 4](image)

**Figure 4.** Engineering stress-strain curves of Fe-23Cr-8.5Ni DSS with a wide range of volume-fraction-weighted grain sizes.

It is interesting to observe that a significant increase in uniform elongation occurs at a structural scale of 375 nm. This is much finer than the scale where a similar transition is observed in single phase metals, which is often above a few micrometers. This observation can be attributed to the unique deformation characteristics of dual phase materials. For DSS, the plastic responses of individual phase to the applied strain are different from each other, resulting in a plastic strain gradient across phase interfaces [13]. This requires extra dislocations to be generated near the interfaces due to geometrical necessity, in order to make allow deformation compatibility [14, 15]. The geometrically necessary dislocations and their development with strain can lead to enhanced work hardening and tensile ductility.

### 4. Conclusions

Fe-23Cr-8.5Ni DSS samples with different structural sizes ranging from the nanometers to micrometers scale have been produced by cold rolling and subsequent annealing. The effect of structural size on the mechanical properties has been studied. The main results are summarized as follows:

- Cold rolling to 90% leads to a significant structural refinement down to 72 nm, which is associated with a high yield strength of about 1.3 GPa but a low tensile elongation of 5%.
- As the structural scale is increased by annealing to 375 nm, a rapid increase in elongation takes place, resulting in a good combination of yield strength and elongation, with values of 738 MPa and 29%, respectively.
- With further increase of the structural scale up to 2.1 \( \mu \)m, the yield strength gradually decreases, but the improvement in tensile elongation is limited.
Acknowledgments
The authors gratefully acknowledge the support from the National Natural Science Foundation of China (NSFC, Grant Nos.51471039, 51327805 and 51671039), and from the Fundamental Research Fund of Central Universities of China (Grant No. 106112017CDJPT130005).

References
[1] Huang X 2009 Scr. Mater. 60 1078-82
[2] Tsuji N, Ito Y, Saito Y and Minamino Y 2002 Scr. Mater. 47 893-9
[3] An H, Wu S, Zhang Z, Figueiredo R B, Gao N and Langdon T G 2012 Scr. Mater. 66 227-30
[4] Li Z, Fu L, Fu B and Shan A 2013 Mater. Lett. 96 1-4
[5] Gao S, Chen M C, Joshi M, Shibata A and Tsuji N 2014 J. Mater. Sci. 49 6536-42
[6] Fang T H, Li W L, Tao N R and Lu K 2011 Science 331 1587-90
[7] Wu X L, Yang M X, Yuan F P, Wu G L, Wei Y J, Huang X and Zhu Y T 2015 Proc. Natl. Acad. Sci. USA 112 14501-5
[8] Koch C C, Scattergood R O and Murty K L 2007 JOM 59 66-70
[9] Fan Z Y, Tsakiropoulos P and Miodownik A P 1992 Mater. Sci. Technol. 8 922-9
[10] Lee S J, Park Y M and Lee Y K 2009 Mater. Sci. Eng. A 519 32-7
[11] Saha R, Ueji R and Tsuji N 2013 Scr. Mater. 68 813-6
[12] Yu C Y, Kao P W and Chang C P 2005 Acta Mater. 53 4019-28
[13] Chen L, Yuan F P, Jiang P, Xie J and Wu X L 2014 Mater. Sci. Eng. A 618 563-71
[14] Ashby M F 1970 Phil. Mag. 21 399-424
[15] Mughrabi H 2001 Mater. Sci. Eng. A 317 171-80