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Parvez Mannan

University of Wollongong, pm999@uowmail.edu.au

Andrii Kostryzhev

University of Wollongong, andrii@uow.edu.au

Elena V. Pereloma

University of Wollongong, elenap@uow.edu.au

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Abstract

The unavoidable phase transformation upon cooling makes a direct observation of austenite microstructure impossible. One of the methods to investigate the high temperature condition of austenite is to use a model alloy, which would not transform to martensite on quenching. In the present work the recrystallisation and grain growth of austenite during thermo-mechanical processing (TMP) were studied for three Ni-30Fe-Nb-C alloys containing 0.33, 0.85 and 1.42 wt% Nb. The austenite microstructures were characterised using optical and scanning electron microscopy, and energy dispersive X-ray spectroscopy. The austenite recrystallisation kinetics was investigated with respect to the precipitate distribution variations. A relative strength of the grain boundary pinning effect from solute Nb and precipitated particles is discussed.

Keywords

composition, thermo, mechanical, processing, microstructure, effect, development, model, ni, 30fe, nb, c, alloys

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Effect of Composition and Thermo-mechanical Processing on Microstructure Development in Ni-30Fe-Nb-C Model Alloys

Parvez Mannan^{1,a}, Andrii G. Kostryzhev^{1,b} and Elena V. Pereloma^{1,2,c}

¹School of Mechanical, Materials and Mechatronics Engineering, University of Wollongong, Wollongong 2522, Australia

²UOW Electron Microscopy Centre, University of Wollongong, Australia

^apm999@uowmail.edu.au, ^bandrii@uow.edu.au, ^celenap@uow.edu.au

Keywords: thermo-mechanical processing, Nb precipitation, recrystallisation, grain growth

Abstract. The unavoidable phase transformation upon cooling makes a direct observation of austenite microstructure impossible. One of the methods to investigate the high temperature condition of austenite is to use a model alloy, which would not transform to martensite on quenching. In the present work the recrystallisation and grain growth of austenite during thermo-mechanical processing (TMP) were studied for three Ni-30Fe-Nb-C alloys containing 0.33, 0.85 and 1.42 wt% Nb. The austenite microstructures were characterised using optical and scanning electron microscopy, and energy dispersive X-ray spectroscopy. The austenite recrystallisation kinetics was investigated with respect to the precipitate distribution variations. A relative strength of the grain boundary pinning effect from solute Nb and precipitated particles is discussed.

Introduction

Grain refinement usually results in simultaneously high strength and toughness of metallic materials [1]. A fundamental step to refine the ferrite grain size in steels is to carefully control the austenite recrystallisation kinetics during TMP. Although a significant amount of research was carried out in this field, a direct observation of the high temperature microstructure is a challenge due to the phase transformation upon cooling to room temperature. Lower temperature transformation products (martensite, ferrite) obscure the high temperature features of microstructure (prior austenite grains and microalloying element precipitates) and complicate investigation of dynamic recrystallisation. To overcome this challenge, Ni-30%Fe can be used as a model alloy for studying the behaviour of austenite at high temperature [2-4]. This model alloy does not undergo phase transformation upon quenching (remains austenitic at room temperature) and has almost similar stacking fault energy and crystallographic texture evolution to those of carbon steel [4].

Niobium is known to retard austenite recrystallisation and refine grain size via the solute drag and grain boundary pinning effects [5-9]. Large Nb-rich particles may enhance sub-grain structure development (formation of twins and deformation bands), which in steels may lead to an increase in the number of ferrite nucleation sites and ferrite grain refinement [10, 11]. In this original paper we study the NbC particle precipitation and austenite grain structure development during TMP of three Ni-30Fe-Nb-C alloys. The effect of alloy composition on the particle size distribution, recrystallisation kinetics and grain growth rate are discussed.

Material and experimental techniques

Three Ni-30Fe-Nb-C (wt.%) alloys (Table 1) in the form of 15 mm thick hot rolled plates were produced and provided by Prof. Hatem Zurob, McMaster University, Canada. NbC dissolution temperatures were calculated to be 976, 1196 and 1355 °C for alloys L, M and H respectively [12]. The plates were cut to standard size samples (20 mm x 15 mm x 10 mm) for TMP simulation in Gleeble 3500. Prior to the main TMP program the recrystallisation stop temperatures, T_{nr} , for the three alloys were determined using analysis of the stress-strain curves obtained during multi-hit compression testing in the temperature range of 1100 - 700 °C. The T_{nr} temperatures were found to be 950, ~1000 and 1040 °C for alloys L, M and H respectively. The main TMP schedule included

(Fig. 1): heating at 1250 °C for 300 s followed by either water quenching or roughing deformation at 1250 °C up to 0.3 strain at 0.1 s⁻¹ strain rate; finishing deformation at 1075 °C up to 1.2 strain at 0.1 s⁻¹ strain rate; followed by either water quenching or holding at 1075 °C during 300 s and water quenching.

Table 1 Chemical compositions of the studied model alloys (% wt)

Alloy Type	Ni	Fe	Nb	C
L	69.7	29.8	0.331	0.040
M	69.2	29.7	0.851	0.114
H	68.8	29.5	1.420	0.187

Fig. 1 Schematic diagram of the TMP.

The heating and roughing deformation temperature of 1250 °C was chosen to be above the NbC dissolution temperatures in alloys L and M, which allowed to assume absence of strain induced precipitation during roughing in these alloys. The finishing deformation temperature of 1075 °C was chosen to be above T_{nr} in alloy H, which assured development of dynamic recrystallisation (DRX) during finishing deformation in all the three alloys. After TMP the samples were cut, polished and etched using a solution of ferric chloride (FeCl₃) and hydrochloric acid (HCl) in water. Optical microscopy of grain structures was carried out using Leica DMRM microscope equipped with Leica Application Suite (LAS) 4.0.0 image processing software. For determination of the grain size distributions about 500 grains were analysed for each TMP condition using Leica Grain Expert software. Imaging of NbC precipitates was carried out using JEOL 7001F FEG scanning electron microscope (SEM). For determination of the particle size distributions 270 – 700 particles were measured for each TMP condition. Energy dispersive X-ray spectroscopy (EDS) of precipitates was carried out using AZtec 2.0 Oxford SEM EDS system. For determination of the particle compositions about 20 particles were analysed for each TMP condition. Vickers microhardness was measured using Struers DuraScan testing machine.

Results

TMP and optical microscopy. The average grain size after reheating at 1250°C decreased from ~500 μm in alloy L to 90 μm in alloy M and to 40 μm in alloy H, i.e. with an increase in Nb content (Figs. 2,a-c). The average grain size after finishing deformation did not vary significantly with Nb content (Fig. 2,d) and was measured to be 19 μm in alloy L, 18 μm in alloy M and 22 μm in alloy H. During holding for 300 s the grain growth took place in all the three alloys (Fig. 2,e). However, it was less pronounced in alloys M and H (the average grain size after holding 27 and 31 μm respectively) compared to alloy L (92 μm).

The flow curves of finishing deformation showed a maximum of stress followed by a softening region for all three alloys (Fig. 3), which is a typical stress-strain behaviour during DRX. Critical and peak stress values of DRX increased from alloy L (101 and 105 MPa) to M (107 and 114 MPa) to H (127 and 130 MPa), i.e. with an increase in Nb content. Absence of a flattened region on the stress-strain curves indicates incomplete DRX for the studied TMP conditions.

SEM of precipitates. In alloy L the SEM imaging and SEM-EDS did not reveal presence of NbC particles for all the studied TMP conditions, which is obviously due to the processing temperatures being above the particle dissolution temperature (976 °C) in this alloy.

Fig. 2 Optical micrographs showing the grain structure in alloys (a) L, (b) M and (c) H after heating; (d) and (e) grain size distributions after finishing deformation and 300 s holding respectively.

Fig. 3 Stress-strain curves obtained during finishing deformation.

However 36 – 140 nm Fe_3C particles of spherical shape precipitated in alloy L during holding after finishing deformation (Fig. 4). In alloys M and H the 40 – 5000 nm NbC precipitates of spherical, ellipsoidal and rectangular shape were observed for all the studied TMP conditions (Fig. 5). Heating did not result in full dissolution of NbC particles in alloy H (Table 2), due to the reheating temperature (1250 °C) being below the particle dissolution temperature (1355 °C) in this alloy. In alloy M almost all the NbC particles dissolved during reheating, due to the reheating temperature (1250 °C) being above the particle dissolution temperature (1196 °C) in this alloy.

Fig. 4 SEM micrographs of alloy L (a) after finishing deformation showing absence of Nb precipitation and (b) after 300 s holding at 1075°C showing Fe_3C precipitation; (c) a typical EDS spectrum of a Fe_3C particle observed in alloy L.

After deformation, the particle number density significantly increased and the average particle diameter decreased in alloy M (Table 2 and Fig. 6), which indicate strain-induced precipitation. In alloy H the particle number density also increased after deformation, however to a significantly

lower extent compared to alloy M. The average particle diameter in alloy H increased after deformation, in contrast to alloy M. These indicate a particle growth in alloy H, which takes place simultaneously with precipitation of new particles. During holding after deformation the particle number density decreased and the average particle diameter increased in both alloys, which indicate the particle growth.

Fig. 5 Representative SEM micrographs showing NbC particles in alloy (a - c) M and (d - f) H after (a,d) holding at 1250°C, (b,e) finishing deformation and (c,f) holding at 1075°C; (g) and (h) are typical EDS spectra of small and large particles respectively.

Discussion

After holding at 1250°C the grain size decreased from alloy L to M to H, i.e. with an increase in the microalloying element content and the NbC number density (Table 2). This indicates a decrease in the grain growth rate following an increase in the grain boundary pinning effect from solute atoms and precipitates, and corresponds to earlier data observed in microalloyed steels [13, 14]. In particular, with an increase in the microalloying element content by $[\text{Nb}+\text{C}]_{\text{M}} / [\text{Nb}+\text{C}]_{\text{L}} = 2.6$ times from alloy L to M the grain size decreased by about 5.5 times. With an increase in the microalloying element content by $[\text{Nb}+\text{C}]_{\text{H}} / [\text{Nb}+\text{C}]_{\text{L}} = 4.3$ times from alloy L to H the grain size decreased by about 12.5 times. Thus, a variation in the grain boundary pinning effect during grain growth was more than 2 times larger than a variation in the microalloying element content leading to this effect.

After deformation the observed grain size was almost similar in all the three alloys, although the NbC number density varied significantly. This could be explained by the following. In alloy L, due to the absence of precipitation, the recrystallization rate was significantly higher than this in alloys M and H. Therefore, several stages of recrystallisation might have taken place in alloy L leading to significant grain refinement (the grain size decreased by about 26 times from the solution

treated to the deformed condition). In alloy M, due to a maximum of the NbC number density, the recrystallisation proceeded at a lower rate but the grain growth was also significantly retarded. Therefore, the grain refinement in alloy M showed a middle value (the grain size decreased by 5 times from the solution treated to the deformed condition). In alloy H, due to a maximum of the microalloying element content, the recrystallization rate was the slowest. Therefore, the grain refinement in alloy H was the lowest (the grain size decreased by 2 times from the solution treated to the deformed condition) and the stress-strain curve (Fig. 3) showed a relatively moderate softening compared to alloys L and M. The recrystallisation retardation in alloy H did not lead to an increased grain size after deformation due to the initial grain size (after heating) being the lowest. A more detailed investigation of DRX in these alloys is required to support these explanations.

Table 2 Dependence of microstructural parameters and microhardness on TMP schedule

Alloy type	[Nb + C], wt %	TMP schedule, °C	Grain size, μm	NbC size, nm	NbC number density, $\times 10^5 \text{ mm}^{-2}$	HV
L	0.371	1250 hold	500 ± 100	no	no	114 ± 7
		1075 deform	19 ± 10	no	no	151 ± 7
		1075 hold	92 ± 20	no	no	124 ± 8
M	0.965	1250 hold	90 ± 20	337 ± 150	0.05	131 ± 10
		1075 deform	18 ± 10	127 ± 10	13.1	184 ± 8
		1075 hold	27 ± 10	217 ± 20	4.2	164 ± 8
H	1.607	1250 hold	40 ± 10	144 ± 50	1.3	146 ± 10
		1075 deform	22 ± 10	204 ± 5	7.2	190 ± 4
		1075 hold	31 ± 10	237 ± 10	6.5	180 ± 7

Fig. 6 NbC particle size distributions for alloys M and H after (a) deformation and (b) holding.

During holding after deformation the grain growth took place, which was accompanied by the particle coarsening and a decrease in the particle number density. The grain growth was the largest in alloy L, due to the absence of precipitation. The grain growth was similar in alloys M and H, although the NbC number density after deformation was higher in alloy M than that in alloy H. Obviously, a larger content of solid solutes and atom clusters, which might be expected in alloy H compared to M, contributed to the grain growth retardation in this alloy.

For all the studied TMP conditions microhardness was higher for a more highly microalloyed material (Table 2), due to grain refinement, solid solution and precipitation strengthening mechanisms. However, the relative strengthening effect after deformation in alloy M compared to L, $\Delta HV_{M-L} / ([Nb+C]_M - [Nb+C]_L) = 55.5$, was higher than this in alloy H compared to L, $\Delta HV_{H-L} / ([Nb+C]_H - [Nb+C]_L) = 31.5$. This may indicate a higher strengthening increment from a larger number of smaller particles (alloy M) compared to a smaller number of larger particles (alloy H), which corresponds to the earlier observed data in microalloyed steels [13]. After holding the microhardness decreased in all the three alloys, due to grain coarsening, recovery of substructure

and particle coarsening. However, the softening in alloy H was lower, compared to M, due to much lower variation in the NbC number density in alloy H.

Summary

Preliminary study of microstructure development in Ni-30Fe-Nb-C alloys during TMP has shown the following. The average grain size in a more highly microalloyed material varied to a lower extent during TMP, due to a decrease in recrystallisation and grain growth rates with an increase in microalloying element content. The critical and peak stresses for DRX increased with an increase in microalloying element content. NbC precipitates were more effective in retarding recrystallisation than Nb and C solute atoms. Precipitate coarsening resulting in a decrease in the grain boundary pinning effect and grain growth, led to a decrease in strength. However, the strength decrease was less pronounced in a more highly microalloyed material.

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