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Abstract

In this original work, the dislocation structure and NbC precipitation were investigated in three Ni-based alloys (70Ni-Fe-0.331Nb-0.040C, 70Ni-Fe-0.851Nb-0.114C and 70Ni-Fe-1.420Nb-0.157C, wt%) thermomechanically processed in the temperature range of 1250-1075 °C. The dislocation structure inhomogeneity (dislocation networks and cell walls), which we observed in the middle and high Nb+C alloys, resulted from the dislocation pile-ups in the vicinity of >200 nm NbC particles. The dislocation density around >200 nm particles exceeded the average values by 5-7 times, and that in the cell walls might exceed the average values by 10 times. Twins and stacking faults were observed in all alloys after solution treatment at 1250 °C, however, they were not observed after 1.2 strain at 1075 °C. The dislocation generation rate during deformation at 1075 °C varied with alloy composition and increased with an increase in the °C, the majority of particles were growing in the high Nb+C alloy, the Nb+C alloy and all the particles were dissolving in the low Nb+C alloy. Deformation to 1.2 strain at 1075 °C resulted in strain-induced precipitation in all alloys and Nb+C alloys.

Keywords

high, nb, temperature, c, model, alloys, dislocation, structure, nbc, precipitation, three, ni, fe

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High temperature dislocation structure and NbC precipitation in three Ni-Fe-Nb-C model alloys

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Abstract

In this original work the dislocation structure and NbC precipitation were investigated in three Ni-based alloys (70Ni-Fe-0.331Nb-0.040C, 70Ni-Fe-0.851Nb-0.114C and 70Ni-Fe-1.420Nb-0.157C, wt. %) thermomechanically processed in the temperature range of 1250 – 1075 °C. The dislocation structure inhomogeneity (dislocation networks and cell walls), which we observed in the middle and high Nb+C alloys, resulted from the dislocation pile-ups in the vicinity of > 200 nm NbC particles. The dislocation density around > 200 nm particles exceeded the average values by 5 - 7 times, and that in the cell walls might exceed the average values by 10 times. Twins and stacking faults were observed in all alloys after solution treatment at 1250 °C, however they were not observed after 1.2 strain at 1075 °C. The dislocation generation rate during deformation at 1075 °C varied with alloy composition and increased with an increase in the < 20 nm particle number density. During cooling in the temperature range of 1250 - 1075 °C the majority of particles were growing in the high Nb+C alloy, the < 20 nm particles were growing in the middle Nb+C alloy, and all the particles were dissolving in the low Nb+C alloy. Deformation to 1.2 strain at 1075 °C resulted in strain-induced precipitation in all alloys and < 20 nm particle growth in the high and middle Nb+C alloys.

Key words: thermomechanical processing, dislocation structure, strain-induced precipitation, Nb carbides, electron microscopy

Introduction

At low processing temperatures (below the recrystallisation stop temperature, T_{nr}) the dislocation structure development significantly influences the room temperature microstructure and mechanical properties in a number of ways: (i) work hardening [1]; (ii) facilitation of grain nucleation for low temperature phases (ferrite [2, 3], bainite or martensite [4]), which leads to grain refinement and transformation strengthening; (iii) dislocation-particle interaction, which leads to precipitation strengthening [5]. At high temperatures (above T_{nr}) the dislocation structure development governs the rates of recovery and recrystallization [6-8], which affect the austenite grain size, high temperature strength, and the load levels on equipment during metal forming operations [9]. After deformation to high strains or following high cooling rates a high temperature dislocation structure can be retained down to lower processing temperatures and may affect the low temperature microstructure. Therefore, understanding of the dislocation structure development at high temperature is of vital importance for efficient control of processing technology and formation of the microstructure and mechanical properties of the final product. Although the dislocation structure development in metals and alloys was thoroughly investigated at ambient temperatures, a direct observation of the high temperature dislocation structure is a challenge. Dislocation annihilation may take place within a few seconds, therefore an accelerated cooling is required to preserve the high temperature microstructure for the following investigation at room temperature. This makes the study extremely expensive in industrial conditions. Phase transformation during cooling (for example austenite to ferrite or martensite in ferritic steels) alters the high temperature microstructure, which significantly complicates extrapolation of the data obtained at room temperature to the high temperature conditions. In addition, the dislocation structure development is sensitive to alloy composition (presence of solute atoms, precipitates, second phases), crystal structure type (fcc, bcc) and thermomechanical processing parameters (temperature, strain and strain rate), which should be kept in mind when comparing the data from various sources. Some key findings of recent years with respect to the dislocation structure development in ferritic (bcc) and austenitic (fcc) steels are presented below.

In ferritic low carbon steel deformed at room temperature a straight lined dislocation structure developed into the irregular structure (containing multiple dislocation-dislocation interactions, knots and tangle) with uniform dislocation density during straining up to 3-4 %; a further strain increase to 5 % led to formation of dislocation clusters (areas of increased dislocation density, pile-ups); and at above 11 % strain the cell structure was observed [10]. In

CrMo-microalloyed steel deformed at room temperature the cell structure was also observed after 11 % strain [11]. In a low carbon steel deformed in the temperature range of 1100 – 900 °C and cooled to room temperature the dislocation substructure varied from a linear (at ~10 % strain) to irregular with tangles (at ~50 % strain) to the cell structure (at ~96 %) [12]. With a decrease in deformation temperature from 900 to 650 °C an increased amount of sub-structure features (serrated ferrite grain boundaries and sub-grain boundaries) were observed in Nb-bearing steel even at quite low strain (10 %) [13]. In Nb microalloyed steel commercially rolled in the temperature range of 880 - 735 °C from 230 mm to 10 mm plate thickness (~95 % total reduction) and cooled to room temperature the dislocation clusters, net arrangements, and start-up of cell walls were observed in some grains, however fully developed cells were not observed [14], which can be a consequence of quite low reduction per pass (below 10 %). In another Nb-microalloyed steel deformed in the temperature range of 900 – 740 °C to 50 % strain the dislocation structure was of irregular type with the average dislocation density increasing with a decrease in deformation temperature [15]. In low carbon steel deformed in the temperature range of 1100 – 900 °C the average dislocation density increased from $\sim 10^{13} \text{ m}^{-2}$ for 10 % strain to $\sim 10^{14} \text{ m}^{-2}$ for 96 % strain [12]. In Nb-microalloyed steel a decrease in finishing deformation temperature from 800 °C to 600 °C resulted in an increase in the dislocation density from $5 \times 10^{13} \text{ m}^{-2}$ to $7 \times 10^{14} \text{ m}^{-2}$ [16]. A decrease in finish cooling temperature was also observed increasing the dislocation density in NbTi microalloyed steel [17]. Therefore in ferritic steels, (i) an increase in strain leads to an increase in dislocation density, due to new dislocation generation, and may result in formation of dislocation tangles and cell walls; and (ii) an increase in temperature leads to a decrease in dislocation density, due to dislocation annihilation, and retards formation of the cell structure, i.e. the cell structure forms at higher strains.

In addition to the strain and temperature effects, the presence of solute atoms and precipitates influences the dislocation structure development. The dislocation density in 0.1 wt. % phosphorus microalloyed steel was observed to be about 1.5 times higher than that in a plain carbon steel processed using the same technology [18]. In Ti-microalloyed IF steel deformed at room temperature to 10 % strain the cell size of dislocation structure was observed decreasing with an increase in TiC volume fraction, which was related to the particle - cell wall interaction [19]. The strain hardening rate during room temperature tensile testing of high strength steel with Nb and Mo additions was higher than that of Ti-microalloyed interstitial free (IF) steel, which was attributed to the dislocation locking by NbMo-rich particles [20]. In microalloyed line pipe steels of various compositions the work hardening rate during room temperature compression

increased with an increase in CuTiNbV-rich precipitate number density and dislocation density, following the restriction of dislocation mobility by the precipitates [21].

In austenitic steels the dislocation structure development follows a similar trend (straight lined structure – irregular structure – tangles – cell walls), however the cell structure forms at higher strains and the deformation twinning may take place. In austenitic 8.3 wt. % Mn steel deformed at room temperature the dislocation tangles and cell structure formation were observed at 20 % strain; twin formation took place after 30 % strain; and deformation bands appeared after 60 % strain (no austenite to martensite transformation was observed in this work) [22]. In 18 wt. % Mn – 18 Cr – 2 Mo – 0.9 N austenitic stainless steel deformed at room temperature the twin formation (in addition to straight lined dislocations) was observed after 5 % strain; the dislocation tangles developed between the twins by 15 % strain and the dislocation cell structure developed by 45 % strain [23]. In 21.2 wt. % Ni – 23.8 Cr – 5.5 Mo – 3.7 Mn – 2.4 W – 0.47 N austenitic stainless steel deformed at room temperature the regular straight lined dislocation structure developed below 5 % strain; at > 5 % strain the twin formation began; however, the dislocation slip dominated as the major deformation mechanism until 35 % strain and the dislocation cell structure appeared by 20 % strain [24]. This behaviour seemed being independent of strain rate up to 2500 s^{-1} , but was dependent on temperature: twin structure developed faster (at lower strains) with a decrease in temperature. In 16.3 wt.% Ni – 10.2 Cr – 2.01 Mo – 0.11 N austenitic stainless steel with an increase in test temperature to $200\text{ }^{\circ}\text{C}$ the dislocation slip was the main deformation mechanism up to 50 % strain; the twin formation was not observed, although some stacking faults (which are considered being the precursors for twinning) were present [25]. In 9 wt. % Ni – 18 Cr austenitic stainless steel deformed in the temperature range of $300 - 800\text{ }^{\circ}\text{C}$ in the strain rate range of $2000 - 6000\text{ s}^{-1}$ up to 50 % strain no twin formation was observed; the dislocation cell structure developed by 15 % strain; with an increase in temperature and a decrease in strain rate the dislocation density decreased and the cell size increased [26]. In 15.8 wt. % Mn – 21.4 Cr – 1.8 Ni – 0.65 N steel deformed at $850\text{ }^{\circ}\text{C}$ the cell structure was also observed after 15 % strain; with an increase in strain the dislocation density increased and the cell size decreased; no twins were observed even after 60 % strain at $850\text{ }^{\circ}\text{C}$. However, twins were present after 60 % strain at $800\text{ }^{\circ}\text{C}$ [27]. Straining of this steel to 60 % at $900\text{ }^{\circ}\text{C}$ resulted in formation of the recrystallised microstructure: grains with a high variation in the dislocation density and recrystallisation twins were observed.

Although precipitates of microalloying elements, in particular NbC, were observed to strongly interact with dislocations in ferritic [19-21, 28, 29] and austenitic [30, 31] steels, and

Fe-Ni-Nb-C model alloys [32-34], a systematic study of the effect of precipitates on dislocation structure development at high temperature has not been carried out. Here we investigate this effect for the first time in three original 70 wt. % Ni-Fe-Nb-C model alloys after different stages of processing in the temperature range of 1200 – 1075 °C. The dislocation structure inhomogeneity has been observed in this work originating from > 200 nm NbC particles, and the dislocation generation rate has been shown here to increase with an increase in the < 20 nm particle number density.

Materials and experimental techniques

Three 70 wt.% Ni-Fe-Nb-C alloys (Table 1) in the form of 15 mm thick hot rolled plates were produced and provided by Prof. Hatem Zurob, McMaster University, Canada. The plates were cut to standard size samples (20 mm x 15 mm x 10 mm) for thermomechanical processing (TMP) in Gleeble 3500. The non-recrystallisation temperatures, T_{nr} , for the three alloys were determined to be 950, ~1000 and 1040 °C for alloys L, M and H respectively [35]. The TMP, carried out for all three alloys according to Figure 1, consisted of 300 s solution treatment at 1250 °C followed by: (i) water quenching, or (ii) roughing deformation at 1250 °C to 0.3 strain at 0.1 s⁻¹ strain rate, cooling at 3 °Cs⁻¹ cooling rate to 1075 °C and water quenching, or (iii) roughing deformation at 1250 °C, finishing deformation at 1075 °C to 1.2 strain at 0.1 s⁻¹ strain rate and water quenching (totally 9 samples). The finishing deformation temperature of 1075 °C was chosen to be above T_{nr} in alloy H, to assure development of dynamic recovery and recrystallisation in all alloys for all TMP conditions. After TMP the samples were studied using optical, scanning electron (SEM) and transmission electron microscopy (TEM). Optical microscopy of the samples is presented elsewhere [35].

Imaging of > 20 nm NbC precipitates was carried out using JEOL 7001F FEG scanning electron microscope operating at 5 kV. SEM sample preparation included polishing with diamond suspensions and slight etching using a solution of ferric chloride (FeCl₃) and hydrochloric acid (HCl) in water. For determination of the particle size distributions, number density and area fraction values up to 700 particles (depending on condition) were measured in alloys M and H. For the > 20 nm particle size range, the number density was expressed in the units of area, μm⁻², as the SEM allows to investigate the samples only in two dimensions. Energy dispersive X-ray spectroscopy (EDS) of NbC particles 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.

Imaging of < 20 nm NbC precipitates and dislocation structure was carried out using thin foils and a JEOL JEM2011 transmission electron microscope operating at 200 kV. The thin foil specimens were prepared using fine polishing on Leica EM TXP machine and ion milling on Leica EM RES101 and Gatan PIPS machines. For determination of the particle size distributions, number density and volume fraction values, 200 – 830 particles were imaged using bright field and dark field TEM imaging techniques for each of 9 studied TMP conditions. For the < 20 nm particle size range, the number density was expressed in the units of volume, μm^{-3} , as the TEM allows to investigate the samples in three dimensions. To verify the precipitate compositions 20 – 25 particles were analysed for each of 9 studied conditions using energy dispersive X-ray spectroscopy (EDS) point analysis on a JEOL TEM EDS system. For the dislocation density determination, 10 - 15 representative regions were imaged for each of 9 studied material conditions using two-beam condition technique [36] avoiding (220) and $(11\bar{1})$ diffraction vector directions, which are conditions of dislocation invisibility in fcc lattice. The foil thickness was measured utilising a convergent beam diffraction technique [36]. The foil thickness was measured to be in the range of 60 – 110 nm with the average value of 70 nm, which was used to calculate the precipitate number density and volume fraction.

Results

SEM and TEM study of NbC precipitates

In alloys H and M, the SEM analysis of precipitates revealed presence of mainly spheroidal and ellipsoidal 20 – 1500 nm NbC particles (Figure 2a) together with some coarse rod-shaped 1500 - 5000 nm NbC particles for all TMP conditions. In alloy L no precipitates were observed by SEM. In alloys H and M, the TEM analysis of precipitates revealed presence of mainly spheroidal 2 – 20 nm NbC particles (Figure 2b) and occasionally ellipsoidal < 300 nm NbC particles for all TMP conditions. The nature of these precipitates was confirmed by the EDS analysis (Figures 2c-h) and TEM diffraction in the following way. The > 50 nm particles (Figure 2c) exhibited a cube-to-cube orientation relationship $((200)_{\text{NbC}} // (200)_{\text{matrix}}, [011]_{\text{NbC}} // [011]_{\text{matrix}}$ in Figure 2d), which is inherent for precipitates with cubic structure in fcc matrix [34, 37, 38]. The prediction of interplanar distances, d-spacing, for NbC can be carried out using equation

$d = a/\sqrt{h^2 + k^2 + l^2}$, where $a = 0.44$ nm [39-43] is the average unit cell size and h , k , and l are Miller indices. The calculations for > 50 nm particles have shown $d_{200} = 0.220$ nm and $d_{111} = 0.254$ nm. The measurements of d -spacing using TEM diffraction patterns have shown $d_{200} = 0.2081$ nm and $d_{111} = 0.2540$ nm, which is in good agreement with the calculated values. In contrast, the < 50 nm particles (Figure 2f) did not exhibit a cube-to-cube orientation relationship ($(220)_{\text{NbC}}// (220)_{\text{matrix}}$, $[111]_{\text{NbC}}// [001]_{\text{matrix}}$ in Figure 2g). Other than cube-to-cube orientations between precipitates with cubic crystal structure and fcc matrix were observed previously [44], however this can be considered being an unusual case which requires further investigation. The calculations of d -spacing for < 50 nm particles have shown $d_{220} = d_{02\bar{2}} = 0.1573$ nm. The measurements have shown $d_{220} = d_{111} = 0.1542$ nm, which is in good agreement with the calculated values. This analysis of diffraction patterns has supported the NbC nature of the observed precipitates.

For all the studied TMP conditions the volume fraction of > 20 nm particles increased with an increase in the Nb+C content in alloy composition, i.e. from alloy L to M to H (Table 2). However, the number density and volume fraction of < 20 nm particles did not show the same trend with alloy composition, which indicates a strong influence of TMP on the particle precipitation kinetics (Figure 3). In the condition after solution treatment the number density of < 20 nm particles increased with an increase in Nb+C content in alloy composition. During cooling after roughing deformation the < 20 nm particle number density decreased and the average particle size increased in alloys M and H, although in alloy L the < 20 nm particle number density did not show a significant variation and the average particle size and volume fraction decreased. In addition, during cooling after roughing the number density of the > 20 nm particles increased in alloys H and M. After finishing deformation the particle number density increased and the average diameter decreased in both particle size ranges in alloys H and M and in the < 20 nm size range in alloy L.

TEM study of dislocation structure

The typical dislocation structure and dislocation density varied with alloy composition and TMP condition (Figure 4, Table 2). After 300 s solution treatment at 1250 °C the straight-lined dislocation structure was observed in all alloys (Figure 4a,d,g); and the average dislocation density slightly decreased with a decrease in Nb+C content in alloy composition (Table 2). Following roughing deformation to 0.3 strain at 1250 °C and cooling to 1075 °C (which has

taken ~60 s) the average dislocation density in alloys H and M was measured to vary insignificantly, compared to the condition after solution treatment (Figures 4b,e); however, the average dislocation density in alloy L increased by more than 7 times (Figure 4h, Table 2). These differences in dislocation density are related to variations in the dislocation structure development in the three alloys. In alloys H and M the dislocations concentrated in clusters and cell walls in the vicinity of > 200 nm particles, which resulted in a decreased average dislocation density. In contrast, the dislocation structure in alloy L was homogeneous with an increased average dislocation density, compared to that in alloys H and M. After 1.2 strain at 1075 °C the typical dislocation structure was of irregular type with knots and tangles in all alloys (Figures 4c,f,i); however, the increase in average dislocation density due to deformation varied with alloy composition and was measured to be 6 times in alloy H, 2.7 times in alloy M, and 3.8 times in alloy L. Deformation twinning or deformation induced dislocation cell structure were not observed in any alloy in any TMP condition, which can be a result of fast recovery and recrystallization [35] experienced by the studied alloys during TMP at relatively high processing temperatures (1250 – 1075 °C), low strain rate (0.1 s^{-1}) and low strain values (0.3 and 1.2 during roughing and finishing deformations respectively).

In addition to the typical dislocation structures presented on Figure 4, the following local substructure arrangements were observed: annealing twins and stacking faults in all alloys after solution treatment (Figure 5a,b); dislocation networks in alloys H and M after solution treatment (Figure 5c); dislocation interactions (bowing and pile-up) with > 200 nm particles (Figure 5d) resulting in an increased dislocation density in the areas around these particles (Figure 5e) in alloys H and M in all TMP conditions; low angle boundaries (Figure 5f); remains of dislocation cells (Figure 5g) and interaction sites of disintegrated cell walls with > 200 nm particles (Figure 5h) in alloys H and M after roughing deformation and cooling to the temperature of finishing deformation; and dislocation interactions (bowing) with small (< 20 nm) particles in all alloys in all TMP conditions (Figure 5i).

Discussion

A key parameter affecting the NbC precipitation in the studied alloys is the Nb+C content. For all the studied TMP conditions, as expected the total particle volume fraction increased with an increase in the Nb+C content, i.e. from alloy L to M to H (Table 2). However, the particle number density did not follow this trend, which reflects the effect of processing. After the

solution treatment the particle number density in both size ranges increased from alloy L to M to H, which is consistent with an increase in the NbC dissolution temperature with an increase in Nb+C content [45]. During cooling after roughing to 1075°C, the particle precipitation kinetics significantly varied with alloy composition (Table 2, Figure 3). In alloy H the average particle diameter and volume fraction increased in both size ranges, which indicate that the growth of all particles was the predominant behaviour in this alloy. The particle number density increased in the > 20 nm size range and decreased in the < 20 nm size range due to growth of < 20 nm particles, i.e. those particles which were calculated within the < 20 nm size range prior to cooling have been calculated within the > 20 nm size range after cooling. Due to the NbC dissolution temperature in alloy H (1355 °C [45]) being above the roughing deformation temperature (1250 °C), some new particles might have precipitated in this alloy during cooling; however, it did not affect the statistical parameters significantly. In alloy M during cooling the average particle diameter decreased and the number density and volume fraction increased in the > 20 nm size range; the average particle diameter and volume fraction increased in the < 20 nm size range, however, the number density decreased in this range. These mean, that in alloy M the growth of < 20 nm particles was the predominant behaviour. In alloy L the < 20 nm particle average diameter and volume fraction decreased during cooling, and the number density did not change significantly. This means that in alloy L the particle dissolution was the predominant behaviour, due to the NbC dissolution temperature in this alloy (975 °C [45]) being significantly below the roughing deformation temperature (1250 °C).

During the finishing deformation the particle precipitation kinetics also varied with alloy composition (Table 2, Figure 3). In alloy H the particle parameters did not vary significantly in the > 20 nm size range, which indicates stability of these particles at the 1075 °C temperature of finishing deformation. In contrast, in the < 20 nm particle size range the average diameter decreased, and the number density and volume fraction increased, which indicates strain induced precipitation. In alloy M the particle average diameter decreased and the number density increased in both size ranges. These mean that in alloy M new particles were precipitating together with some < 20 nm particles growing to the size of > 20 nm. The lowest < 20 nm particle number density observed after deformation in alloy M, compared to two other alloys, is compensated by the highest > 20 nm particle number density in this alloy. In alloy L the particle average diameter decreased, and the number density and volume fraction increased, which means that the new NbC precipitation was the predominant process in this alloy.

A key parameter affecting the dislocation structure variation in the studied alloys is the variation in NbC precipitation, in particular, presence of large (> 200 nm) particles in alloys H and M and their absence in alloy L. The interactions of moving dislocations with large (> 200 nm) particles in alloys H and M resulted in the dislocation structure inhomogeneity in these alloys, compared to a relatively uniform dislocation structure in alloy L. The dislocation density was 5 - 7 times higher in the areas around large (> 200 nm) particles, compared to the average values. Complexity of the dislocation structure around large particles was mentioned in a number of works [46-49] as a source of grain nucleation during recrystallization in aluminium, nickel and steel. However, direct measurements of the dislocation density around large particles are scarce [50]. The dislocation cell walls, with an order of magnitude higher dislocation density than the average values, were observed in alloys H and M, although they were not present in alloy L. The appearance of cell walls was associated with the presence of large particles. The dislocation pinning by large particles leading to the dislocation pile-ups around them, which subsequently developed into the cell walls, could be suggested as a possible mechanism of the cell-wall formation.

After solution treatment the dislocation structure was more or less homogeneous and looked similar in all alloys. The average dislocation density variation for this TMP condition (Table 2) followed the trend for the particle number density: an increase in the average dislocation density from alloy L to M to H corresponds to an increase in the particle number density. This can be explained by an increase in the number of potential dislocation-particle interaction sites with an increase in the particle number density, leading to the retardation of dislocation annihilation in a more highly alloyed alloy.

Following 60 s cooling period after roughing deformation to the temperature of finishing deformation the dislocation structure and average dislocation density significantly varied for alloys H and M compared to L. In alloys H and M the dislocation structure was inhomogeneous and the average dislocation density was lower, compared to alloy L in which the dislocation structure was relatively homogeneous and the average dislocation density was higher. These can be explained by the following. During cooling the dislocation motion was restricted by large (> 200 nm) particles in alloys H and M to a greater extent compared to alloy L containing only small (< 20 nm) particles. Following partial annihilation and polygonisation, formation of the cell walls and sub-grain boundaries took place in alloys H and M in the vicinity of large particles (Figure 5f,g,h), although it did not happen in alloy L (Figure 4h). The average dislocation density was measured at a distance from the areas of high dislocation concentrations in alloys H and M.

Therefore, the values of average dislocation density were lower in alloys H and M than those in alloy L (Table 2).

During the finishing deformation the average dislocation density increased in all alloys following generation of new dislocations (Table 2); however, the relative and absolute increments in dislocation density varied with alloy composition. The relative increase in dislocation density followed a variation in the < 20 nm particle number density: the highest in alloy H, and the lowest in alloy M. This indicates the role of small particles in development of dislocation structure: immobilisation of dislocations by small particles enhances the generation of new dislocations. An enhanced work hardening due to presence of small particles was observed earlier in some ferritic steels [21, 51]. The highest absolute increase in the average dislocation density was observed in alloy L, which can be explained by a more homogeneous dislocation distribution in this alloy due to the absence of large particles.

Schematically the dislocation structure development in the studied alloys is summarised in Figure 6. In alloys H and M the cell walls appeared during cooling after roughing deformation following the dislocation pinning by large (> 200 nm) particles; in contrast, in alloy L the dislocation distribution was more homogeneous and the wall formation did not occur. The average dislocation density after finishing deformation was higher in alloys H and L, compared to alloy M, due to a larger number density of small (< 20 nm) particles in these alloys leading to an enhanced dislocation pinning and generation of new dislocations.

Conclusions

Investigation of the NbC precipitation and its effect on dislocation structure development in three original 70 wt.% Ni-Fe-Nb-C alloys thermomechanically processed in the temperature range of 1250 – 1075 °C has shown the following:

1. After the solution treatment at 1250 °C the total NbC number density and volume fraction increased with an increase in the Nb and C contents in alloys composition. The average dislocation density increased with an increase in the precipitate number density, which is related to an increase in potential number of dislocation-particle interaction sites, enhanced dislocation immobilisation, and retardation of annihilation. Annealing twins and stacking faults have been observed in all alloys after the solution treatment.
2. The NbC precipitation kinetics during cooling after deformation at 1250 °C significantly varied with alloy composition. The predominant process in the high Nb+C alloy 70Ni-Fe-

- 1.420Nb-0.157C was the growth of particles in all size ranges, although some precipitation of new particles might have taken place. The predominant process in the middle Nb+C alloy 70Ni-Fe-0.851Nb-0.114C was the growth of < 20 nm particles. All particles were dissolving in the low Nb+C alloy 70Ni-Fe-0.331Nb-0.040C. These types of behaviour are related to a decrease in the NbC dissolution temperature with a decrease in Nb and C contents. During cooling after roughing the dislocation interactions with > 200 nm precipitates resulted in formation of the dislocation networks, cell walls and sub-grain boundaries in the alloys with high and middle Nb+C content. This led to development of an inhomogeneous dislocation structure with a decreased average dislocation density in the alloys with high and middle Nb+C content, compared to a more homogeneous dislocation structure with an increased average dislocation density which developed in the alloy with low Nb+C content.
3. During deformation to 1.2 strain at 1075 °C the strain-induced precipitation took place in all three alloys, with some < 20 nm particles growing in the alloys with high and middle Nb+C content. The irregular dislocation structure developed in all alloys. The dislocation generation rate varied with alloy composition. An increased dislocation generation rate corresponded to an increased number density of < 20 nm particles. No deformation twins or stacking faults have been observed in any alloy after the deformation.

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Table. 1 Chemical composition of the studied alloys (wt. %)

Alloy	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

Figure 1. The schematic diagram of thermo-mechanical processing.

Figure 2. Representative (a) SEM image, (b) bright field TEM image and (c) dark field TEM image of NbC particles (alloy H after deformation at 1075 °C); selected (d) TEM image, (e) diffraction pattern and (f) TEM EDS spectrum of large NbC particle (alloy H); selected (g) TEM image and (h) diffraction pattern of small NbC particle; (i) TEM EDS spectrum of the matrix (alloy H).

Table 2. Variation in the parameters of NbC particles and dislocation density with alloy type and processing schedule

alloy	Size range, nm	Average diameter, nm			Number density*			Volume fraction**			Dislocation density, $\times 10^{13} \text{ m}^{-2}$		
		ST [#]	BD	DEF	ST	BD	DEF	ST	BD	DEF	ST	BD	DEF
H	< 20	3.4 ± 1.0	5.5 ± 2.0	2.5 ± 0.8	16964	6452	74750	0.00046	0.00074	0.00090	1.2 ± 0.5	1.0 ± 0.4	6.0 ± 0.8
	> 20	144 ± 50	224 ± 200	204 ± 150	0.132	0.577	0.717	0.00452	0.05010	0.03954			
M	< 20	2.9 ± 0.9	3.2 ± 1.5	2.7 ± 1.2	16153	13880	24333	0.00028	0.00044	0.00048	0.9 ± 0.6	1.2 ± 0.3	3.3 ± 0.6
	> 20	337 ± 150	268 ± 200	127 ± 90	0.005	0.191	1.111	0.00209	0.02218	0.02015			
L	< 20	4.8 ± 1.6	2.9 ± 1.1	2.6 ± 0.9	10227	10680	38000	0.00085	0.00022	0.00047	0.6 ± 0.1	4.5 ± 0.6	17.2 ± 0.1
	> 20	no	no	no	no	no	no	no	no	no			

[#]ST, BD and DEF are sample conditions: ST - after solution treatment at 1250 °C, BD - before finishing deformation at 1075 °C, and DEF - after finishing deformation at 1075 °C;

* unit of the particle number density is μm^{-3} and μm^{-2} for < 20 nm and > 20 nm particles, respectively;

** the area fraction of particles, measured using SEM imaging, can be assumed equal to the volume fraction of particles for the high number of particles studied in this work

Figure 3. Precipitate size - number density distributions for three studied TMP conditions in alloys (a) H, (b) M and (c) L.

Figure 4. Typical bright field TEM images (all close to $[001]$ zone axis) of dislocation structure in alloy (a, b, c) H, (d, e, f) M, and (g, h, i) L; (a, d, g) after solution treatment, (b, e, h) before deformation at 1075 °C, and (c, f, i) after 1.2 strain at 1075 °C.

Figure 5. Selected TEM images of (a) annealing twins and (b) stacking faults observed in all alloys after 300 s solution treatment at 1250 °C (a and b are alloy H); (c) dislocation networks in alloys H and M after 300 s solution treatment at 1250 °C (c is alloy M); (d) dislocation interactions with large particles and (e) local dislocation structure in the vicinity of large particles in alloys H and M in all TMP conditions (d and e are alloy H after solution treatment); (f) low angle boundaries, (g) remains of dislocation cells, and (h) interactions of disintegrated cell walls with large particles in alloys H and M after roughing deformation at 1250 °C to 0.3 strain and cooling at 3 °Cs⁻¹ cooling rate to 1075 °C (f, g and h are alloy H); (i) dislocation interactions with small particles in all alloys in all TMP conditions (i is alloy L after deformation).

Figure 6. Schematic presentation of the dislocation structure development in the studied alloys.