Note

Microstructural Characterization of Precipitation in an Isothermally Aged Nb-containing Microalloyed Steel

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In order to control the grain size by the addition of microalloying elements, it is important to realize that the sequence of casting, reheating, control rolling and subsequent weld cycles may produce a complex series of precipitation reactions.¹⁾ To characterize these reactions in a quantitative manner, it is essential to attempt to define the structure, morphology, composition, size and distribution of precipitate phases produced during the thermal processes. The role of precipitate particles is to refine austenite grains by the pinning of the boundaries during normalizing heat treatments or during thermomechanical working, and to harden the ferrite grains on cooling after transformation.^{1,2)}

The goal of present work is to characterize chemically, structurally and microstructurally the precipitate phases formed in a Nb-containing microalloyed steel during isothermal aging by means of X-ray diffraction (XRD), scanning and transmission electron microscopes (SEM and TEM) and EDS microanalysis.

The chemical composition of microalloyed steel was as follows: 0.045% C, 0.425% Mn, 0.008% Si, 0.012% P, 0.021% Nb, 0.0041% N and 0.043% Al, expressed in mass percent. Samples of about 1 cm×1 cm×1 cm were cut and austenitized at 1 120°C for 30 min and subsequently cooled down to 800, 850, 900, 950, 1 000 and 1 050°C for isothermal transformation for 10 to 10 000 s and then water-quenched. Isothermally heat-treated samples were prepared metallographically and etched with Nital reagent for their observation with a scanning electron microscope at 20 kV. Precipitates were extracted by an electrolytic dissolution of the ferrite matrix in a solution composed of 10 vol% HCl in water at 3 V (AC). The extracted residues were analyzed by X-ray diffraction with a monochromated K α copper radiation. The TEM specimens were prepared employing the twin-jets polishing technique with an electrolyte composed of 10 vol% perchloric acid in methanol at –40°C at 50 V. Specimens were observed in a transmission electron microscope at 200 kV. Microanalysis of precipitates was performed in an EDS equipment.

Figures 1(a)–1(d) show the SEM micrographs of samples transformed isothermally at 850°C for 1 and 100 ks, and at 950°C for the same times, respectively. An intragranular precipitation on ferrite grain boundaries is observed for all cases. Widmanstatten ferrite and bainite were found in the samples aged at 850 and 950°C, respectively, for 1 ks because of the austenite transformation during cooling after isothermal aging. The samples aged at 850 and 950°C for 100 ks showed mainly the presence of equiaxial ferrite and intragranular precipitates. The Time–Temperature–

Fig. 1. SEM micrographs of samples aged at 850°C for (a) 1 and (b) 100 ks, and at 950°C for (c) 1 and (d) 100 ks.

Fig. 2. TTP diagram for the microalloyed steel.

Fig. 3. XRD patterns for the residues extracted from samples aged at (a) 850°C and (b) 950°C for 100 ks.

Precipitation (TTP) diagram for both the intragranular and transgranular precipitations of aged samples, determined from SEM observations, is shown in Fig. 2. As expected, the intragranular precipitation preceded to the transgranular one. The fastest precipitation kinetics was observed to occur for agings at 950°C. This fact is in agreement with other TTP diagrams for similar microalloyed steel compositions reported by other authors.²⁻⁵⁾

Figures 3(a) and 3(b) illustrate the X-ray diffraction patterns of the residues extracted from samples aged at 850 and 950°C for 100 ks. The AlN and NbC phases were mainly detected in these residues. The XRD patterns of residues for samples aged at other temperatures also showed the same phases.

The BF-TEM micrograph for two precipitates, its corresponding electron diffraction pattern and precipitate EDX spectrum for the sample aged at 900°C for 100 ks are shown in Figs. 4(a)–4(c), respectively. The TEM image

Fig. 4. (a) BF-TEM micrograph, (b) electron diffraction pattern with an axis zone, $B=[011]$ for an fcc structure and (c) EDX spectrum for a NbC precipitate.

Fig. 5. (a) BF-TEM micrograph, (b) electron diffraction pattern with an axis zone, $B=[2\bar{1}10]$ for a cph structure and (c) EDX spectrum for an AlN precipitate.

shows that the precipitates have a rounded shape. The indexing of electron diffraction pattern indicated that these precipitates correspond to the NbC phase. Besides, The EDX spectrum illustrates the characteristic radiations for C and Nb.

Figures 5(a)–5(c) show the BF-TEM image, electron

diffraction pattern and EDX spectrum for a precipitate, respectively, in the sample aged at 900°C for 100 ks. The morphology of this transgranular precipitate is like a plate. The electron diffraction patterns indicates that the precipitate correspond to an AlN phase, which is also confirmed by the presence of Al and N characteristic radiations in the EDX spectrum.

The above results indicated that the precipitate phases are mainly AlN and NbC in aged samples. This fact was verified using the prediction method for precipitate phases, in microalloyed steels containing Nb, C, N and Al, proposed by Keown and Wilson.⁶⁾ Their method is based on the application of the thermodynamic concept of “lines of equal solubility” for the different compounds. For example, they showed that the AlN phase is stable at 900°C for a Nb content between 0.007 and 0.07%, and for an Al content between 0.01 and 0.1%. On the other hand, the NbC phase is stable at the same temperature for a N content between 0.001 and 0.007%, and for a C content between 0.015 and 0.1%. Thus, it was determined that the AlN and NbC compounds were the most stable phases at 900°C for the steel composition of this work. The predicted precipitate phases agree with the observed ones in aged samples.

It is important to mention that the microstructural characterization of isothermally aged microalloyed-steels is a good alternative to follow the precipitation reactions and enables us to figure out the TTP diagrams, which can be used to describe the plastic behavior or mechanical properties of this type of steel during or after its processing.

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