Influence of Critical Carbide Dissolution Temperature during Intercritical Annealing on Hardenability of Austenite and Mechanical Properties of DP-980 Steels

Calixto Isaac GARCIA,1) Kengun CHO,1) Konstantin REDKIN,1) Anthony John DEARDO,1,3) Susheng TAN,2) Mahesh SOMANI3) and Leo Pentti KARJALAINEN3)

1) Basic Metals Processing Research Institute, Department of Mechanical Engineering and Materials Science, University of Pittsburgh, Pittsburgh, PA 15261, USA. 2) NanoScale Fabrication and Characterization Facility, Petersen Institute of NanoScience and Engineering, University of Pittsburgh, Pittsburgh, PA 15261, USA. 3) Department of Mechanical Engineering, University of Oulu, P.O. Box 4200 (Linnanmäki), FIN-90014, Finland.

(Received on December 20, 2010; accepted on March 17, 2011)

The hardenability of intercritically formed austenite and the mechanical properties of DP-980 steels were found to be strongly influenced by the state of the dissolution reaction of carbides during intercritical annealing. The carbide dissolution behavior of 0.15C–Nb–Cr–Mo–V steels was investigated.

KEY WORDS: austenite formation; carbide dissolution; dual-phase; intercritical annealing temperature; CGL.

1. Introduction

Since the introduction of Advanced High-Strength Sheet Steels (AHSSs), automakers have taken advantage of these special types of steels to promote the production of green vehicles. These new vehicles will have lightweight materials, which improve fuel efficiency and decrease environmental pollution. In addition, the use of AHSSs has enabled automakers to improve passengers’ safety, apply well-established and proven technologies, and fabricate vehicles at reasonable prices. One of the areas that receives considerable attention in the development of AHSSs is the application of novel processing conditions to optimize the microstructure, increase the properties and reduce the cost of high strength dual-phase (DP), transformation induced plasticity (TRIP), complex-phase (CP), and martensite (MART) steels being produced in Continuous Galvanizing Lines (CGL). For example, the production of high strength 980 DP steels utilizes diverse alloy design philosophies and processing conditions ranging from relatively basic approaches, such as: controlling the annealing cycle (intercritical annealing temperature and cooling speed) to attain the desired dual phase (%$\alpha$ + %$\alpha'$) microstructure by suppressing the bainitic transformation during cooling;1–5) thermomechanical processing in the intercritical region;6) producing ultrafine grained $\alpha + \alpha'$ DP steels via equal channel angular pressing;7) a relatively new heat treatment referred as the Quench Partitioning approach to produce AHSSs.8,9) Recent work10) conducted at the Basic Metals Processing Research Institute (BAMPRI, University of Pittsburgh) has shown that the combined alloy design of 0.15 wt% C–Mn–Cr–Mo–Nb–V steels and the application of a straightforward heat treatment cycle that promotes the full dissolution of carbides during intercritical annealing are very effective in increasing the hardenability of the intercritically formed austenite. The importance of increasing the hardenability of the austenite prior to its decomposition implies that considerably lower cooling rates can be used during the cooling stage in the CGL line. This condition makes unnecessary to cool the strip to very low quench end temperatures to form the expected dual phase microstructure. The major objective of this work was to define the optimal intercritical annealing temperature to achieve full-dissolution of carbides for the steel compositions used in this study. The experimental design and detailed microstructural characterization of the steels were performed by implementing the theoretical predictions from the JMat-Pro thermodynamic software, rapid-heating annealing simulations, and SEM, TEM observations. The CGL simulations of intercritical annealing heat treatments were conducted using the Gleeble 3500 system. The thorough microstructural characterization and the resulting mechanical properties observed in this study will be presented and discussed in this paper.

2. Experimental Procedure

Melting and processing. The chemical composition of the steels used in this study is shown in Table 1. Steel DPTR0 is a Nb-bearing steel and represents the base steel. Steel DPTR1 is the base steel + V addition. The laboratory heats were melted in a vacuum induction-melting furnace. The ingot size was 550 mm (in length) $\times$ 210 mm (in width) $\times$ 210 mm (in thickness). The laboratory ingots were then
reheated at 1200°C for 2 hours and subsequently hot rolled to a final thickness of 3 mm. The finish rolling and coiling temperatures were 920°C and 550°C, respectively. The hot band material was cold rolled 60% to achieve a final thickness of approximately 1.4 mm. The details of the thermo-mechanical processing (TMP) used in all laboratory steels are illustrated in Fig. 1. Samples after cold rolling were sectioned from the two steels and then subjected to a series of experiments including: i) the study of the carbides dissolution and the formation of austenite during intercritical annealing; ii) the behavior of austenite decomposition during cooling from the intercritical annealing temperatures; iii) the estimation of the mechanical properties.

2.1. Intercritical Annealing

The theoretical prediction of the austenite formation and its equilibrium solid solution composition during intercritical annealing was conducted using the JMatPro thermodynamic and kinetic software package. In addition, this software was also used to predict the intercritical temperature to attain different volume fraction combinations of $\alpha + \gamma$, as illustrated in Table 2. Previous work has shown that a phase balance of approximately 50% $\alpha + 50\% \gamma$ is necessary to attain ultimate tensile strength (UTS) equal to 980 MPa. Samples (10 × 5 × 1.4 mm) in size cut from the steels in the cold rolled condition were used to investigate the carbide dissolution behavior during intercritical annealing. The intercritical annealing heat treatments were conducted with the aid of the computer controlled high-speed furnace. The furnace was operated using similar processing conditions of a typical CGL, i.e., a heating rate of 3°C/sec and 60 seconds holding time at the given intercritical annealing temperature.

This part of the study was conducted to define both the dissolution temperature of Fe$_3$C and alloyed carbides present in the starting microstructure of the steels being investigated and to establish the optimum intercritical annealing temperatures to obtain the desired $\alpha + \gamma$ phase balance for the experimental steels: DPTR0 and DPTR1.

2.2. CGL Simulation Annealing Studies

The results obtained from the intercritical annealing experiments were used to design the CGL simulation annealing studies which were conducted with the aid of a Gleeble 3500 system. The intercritical annealing temperatures were selected to obtain the desired amount of $\alpha + \gamma$ and then quenching to the quenching end temperature, $(T_q)$, i.e., 460°C or 350°C, held for 15 seconds, and then fast cooling to room temperature (FCRT) and/or air cooling to room temperature (ACRT), see Fig. 2. Samples from these Gleeble experiments were sectioned to conduct microstructural analysis and tensile tests.

2.3. Hardness Evaluation

The bulk hardness and microhardness evaluation of each resulting ferrite ($\alpha$) and martensite ($\alpha'$) phases after cooling from the intercritical temperature experiments were evaluated and recorded with the aid of a LECO (VHN) hardness tester using a load of 300 g and with a Micro Indentation tester (MHT) using a load of 5 g, respectively. The MHT system has a load range from 1 g to 300 g.

2.4. Microstructural Analysis

The microstructural analysis of all the intercritically annealed samples was conducted using standard optical microscopy (OM) and electron optic techniques such as
scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The first level of microstructural analysis and the corresponding volume fraction of austenite (observed as martensite) and ferrite were assessed using high resolution Keyence (× 5,000) and Nikon optical microscope systems with an automated NOVA-IV Prime image analysis system. The second level of microstructural analysis was conducted using a Philips XL-30 FEG-SEM microscope. The third level of microstructural analysis was conducted using a JEOL-2100F (TEM) at an operating voltage of 200 KV. The composition of the carbides was analyzed using an EDS-Oxford system which is attached to TEM microscope. The samples for TEM and HRTEM analysis were cut in cross-section at an inclined angle to the rolling surface using the diamond precision saw. The cross-section specimens were polished using grinding papers to make both surfaces of the specimens parallel to each other, and then chemically thinned to 100 μm. Finally, thin foil specimens for TEM and HRTEM were electrochemically polished on a conventional twin-jet polisher at room temperature using the 90% Acetic acid and 10% perchloric acid and the polishing was run at 35 V and 70–90 mA.

2.5. Mechanical Testing
The mechanical properties of the specimens resulting from the CGL simulation treatments were evaluated using a computer-controlled MTS 880 servo-hydraulic machine. Two ASTM sub-size tensile specimens (25.4 mm nominal gage and 6.4 mm nominal width) per condition were tested at a cross-head speed of 2 mm/min. The final total elongation was corrected for the reduced gage length using standard area correction calculations.

3. Results and Discussion
3.1. Dissolution Behavior of Carbides
The results of this study clearly showed the importance of establishing the optimum dissolution temperature of carbides and its effect on the carbon content of the intercritically formed austenite. That is, the carbon content from the dissolution of the carbides increases the hardenability of the austenite. The net effect of this behavior is an increase in the type and consequently the hardness of the martensite resulting from the decomposition of austenite during cooling. Figure 3 shows the $\alpha + \alpha'$ dual-phase microstructure of steel DPTR0 which was treated by using either the rapid speed furnace or the Gleeble system with the intercritical annealing temperature of 740°C. From this figure it is clear that there are many undissolved carbides (Fe$_3$C) and alloyed (Cr,Mo,Nb,V) in ferrite. Due to these undissolved carbides, the hardenability of the $\gamma$ is lower than expected and predicted by the thermodynamic software package JMat-Pro, hence resulting in lower hardness (strength) values.

Figure 4 shows the hardness results from the bulk and from each of the individual phases $\alpha$ and $\alpha'$ for the steels used in this study. The results presented in Fig. 4(a) indicates two important points: 1) a large variability in the VHN hardness value with increasing intercritical annealing temperature, i.e. as the %$\gamma$ increases; 2) the hardness values become more stable as the intercritical temperature approaches the dissolution temperature of the Fe$_3$C carbides, in this case at about 770°C, for steel DPTR0, and 780°C, for steel DPTR1. The interesting part of these results is that between 740°C and 765°C the bulk hardness of the dual-phase microstructure does not show the expected continuous increase in hardness with increasing amounts of $\gamma$ or the volume fraction of the composite ($\alpha + \alpha'$). An argument can be made that the variation in hardness values might be directly associated with the local variation of the microstructure. As the intercritical temperature increases above 760°C the variability in the hardness values seems to decrease and the behavior is more conventional, that is, higher VHN hardness values as the amount of $\gamma$ increases. However, another possible explanation regarding the variability in the bulk hard-
ness behavior could also be explained by the presence of undissolved carbides at the lower intercritical annealing temperatures. As the stability of the system with respect to the bulk hardness increases, the amount of undissolved carbides decreases. The effect of the dissolution of the carbides with increasing intercritical temperatures on the hardness of the individual microconstituents (α) and (α′) is shown in Fig. 4(b). This Figure shows the hardness of both α and α′ increasing with intercritical annealing temperatures. These results are very interesting, because based on equilibrium thermodynamic calculations the carbon content in the austenite should decrease with increasing the intercritical annealing temperature, see Fig. 5. The microhardness results presented in Fig. 4(b) clearly show that the hardness of α increases accordingly with the dissolution of the carbides. This implies that the local carbon concentration of the austenite formed intercritically is much higher than the predicted carbon content in equilibrium with γα from the phase diagram. The increase in the hardness of α is not quite understood at this time, it can simply be related to the increase in the local dislocation density produced by the formation of harder α′ during cooling. The overall results from the intercritically annealed experiments showed that the dissolution temperature of the carbides in steel DPTR0 was 770°C, while in steel DPTR1 was 780°C, see Fig. 6. The difference in the dissolution temperature of carbides between the two steels is most likely associated with the combined effect of Nb+V+Mo+Cr on the diffusion of carbon in austenite.

3.2. Microstructural Analyses

The fine detailed microstructural analysis was conducted using thin foils prepared from the intercritically annealed samples. Figure 7 shows a bright field TEM micrograph of twinned α and the presence of Fe3C carbides in a sample intercritically annealed at 760°C and rapidly cooled to 460°C and then ACRT. The presence of twinned α is an indication of the relatively high carbon content of the austenite prior to transformation.

Figure 8 shows another TEM micrograph (BF and DF) of the presence of twinned α in the final α+α′ microstructure. The twinned α contains retained austenite. The change of lath α to plate martensite as the carbon content of the γ increased might have been prevented by the presence of Si, Cr, V, and Mo. The presence of these ferrite stabilizers is known to favor the transition from lath to twinned α.12) The lower the intercritical annealing temperature (below the dissolution temperature of the carbides), the phase balance observed in the resulting microstructure was identified as typical lath α, some areas with twinned α and the presence of undissolved Fe3C carbides in the α matrix. Attempts to determine the tetragonality of the martensite (c/a lattice parameter ratio) as the carbon content of the intercritically formed γ increased due to the dissolution of the carbide particles were conducted using high resolution transmission electron microscopy (HRTEM). A typical example of these observations and measurements is illustrated in Fig. 9. This
HRTEM micrograph corresponds to the identification of twinned $\alpha'$, the measured c/a was 1.027 which corresponds approximately to a carbon content of the $\gamma$ of about 0.60 wt%. Measurements of other twinned $\alpha'$ observed after transformation of $\gamma$ from higher intercritical annealing temperatures had c/a ratios of 1.05 which correspond to $\gamma$ having a carbon content of about 1.1 wt%.

### 3.3. Mechanical Properties

The results of the mechanical properties after the CGL simulation treatments in the Gleeble 3500 system for steel DPTR0 are presented in **Table 3**. The intercritical annealing temperatures selected in this part of the study were based on the results shown in Fig. 4. Two annealing temperatures were selected 740 and 770°C. In addition three quenched end temperatures ($T_Q$) were also used. These were selected from the thermodynamic predictions of the start transformation temperature of bainite (Bs), martensite (Ms) and 50% martensite transformation temperature (M50) calculated using JMat-Pro. The resulting mechanical properties are shown in Table 3. Regardless of the $T_Q$ the UTS of all the samples processed using 740°C as the intercritical annealing temperature did not achieve the 980 MPa value. On the other hand, when the intercritical annealing temperature was increased to 770°C and quenched to various $T_Q$ temperatures, the measured UTS value exceeded 1200 MPa with
good ductility. These results are very attractive because it shows that UTS values in excess of 1200 MPa can be obtained with relatively low carbon content in the steel bulk composition (0.15C wt%). In addition, the results presented in Table 3 also show that there is no need to use very low T₀ temperatures to obtain an UTS value of 1200 MPa. A comparison of the mechanical property results between the samples annealed at 740°C (undissolved carbides) or at 770°C (all carbides dissolved) clearly indicates the influence of the dissolution of carbides as function of the intercritical annealing temperature on the hardenability of austenite.

4. Conclusions

From the results of this study, several conclusions can be drawn: 1) the dissolution temperature of Fe₃C or alloyed carbides cannot be predicted accurately by thermodynamic equilibrium software programs; 2) the hardenability of austenite can be increased by the carbon content from the dissolution of the carbides during intercritical annealing; 3) local carbon concentration in austenite in the intercritical region was calculated based on the tetragonality of the martensite between 0.6 and 1.1 wt%; 4) this local carbon enrichment of the austenite is sufficient to produce a DP steel with UTS higher than 1200 MPa and total elongations of about 13% without having to use very low T₀ temperatures.

Acknowledgements

The authors would like to thank Vanitec Corporation for their financial support of this program. In addition the authors would like to thank BAMPRI, the MEMS Department and the NanoScale Fabrication and Characterization Facility, Petersen Institute of NanoScience and Engineering at the University of Pittsburgh and the University of Oulu, Finland for providing the facilities to conduct this work.

REFERENCES

1) G. R. Speich: Fundamentals of Dual-Phase Steels, ed. by R. A. Kot and B. L. Bramfit, TMS-AIME, Warrendale, PA, (1981), 3.
2) Y. Futamura and M. Miura: Kobelco Technology Review, 28 (2008), 3.
3) D. T. Llewellyn and D. J. Hillis: Ironmaking Steelmaking, 23 (1996), 471.
4) K. Cho, C. I. Garcia, H. Shu, T. R. Chen and A. J. DeArdo: Proc. Int. Conf. on Microalloyed Steels: Proc., Microstructure, Properties and Performance Proc., AIST, Warrendale, PA, (2007), 313.
5) O. Kwon, K. Lee, G. Kim and K. Chin: Mater. Sci. Forum, 638–642 (2010), 136.
6) E. Ahmad, T. Manzoor and N. Hussain: Mater. Sci. Eng. A, 508 (2009), 259.
7) Y. I. Son, Y. K. Lee, K. T. Park, C. S. Lee and D. H. Shin: Acta Mater., 53 (2005), 3125.
8) J. Speer, D. K. Matlock, B. C. De Cooman and J. G. Schroth: Acta Mater., 51 (2003), 2611.
9) E. De Moor, S. Lacroix, A. J. Clarke, J. Penning and J. G. Speer: Metall. Mater. Trans. A, 39A (2008), 2586.
10) K. Cho, K. Redkin, C. I. Garcia and A. J. DeArdo: Technical report to VANITEC, BAMPRI, University of Pittsburgh, Pittsburgh, PA, (2010).
11) J. H. Bucher and E. G. Hamburg: Formable HSLA and Dual-Phase Steels, Metallurgical Society of AIME, Warrendale, PA, (1979), 142.
12) A. K. Sinha: Physical Metallurgy Handbook, ed. by McGraw-Hill, New York, (2003), 8.1.