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Ultrafine Grain Refinement in a Low Alloy Steel

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The phenomenon of grain refinement was studied in a steel containing 0.15% C, 0.32% Si, 1.4% Mn, and 0.43% V. Initial austenite grain size was found to be 50 μm, determined by quenching the specimen in an iced brine solution from 1150 °C. Transformational grain refinement (TGR) was applied to give a reasonable refinement in the grain size. A rolling reduction of about 67% was given to specimens at 900 °C, which was followed by air cooling. Cold rolling and recrystallization of these specimens gave refinement of grains down to 1 μm size was obtained. The electron backscattered diffraction (EBSD) technique was used to determine low- and high-angle grain boundaries that are effectively used to determine the substructure contribution at various stages of recrystallization.

Keywords recrystallization, ultrafine grain refinement, V microalloyed steel

1. Introduction

There has been a considerable interest to produce steels with more refined ferrite microstructure than conventionally produced by controlled rolling, to improve the tensile properties of steel. The refined microstructure of the steel can be correlated with tensile yield strength by the well-known Hall-Petch relationship as follows:

$$\sigma_y = \sigma_0 + k_y d^{-1/2} \quad \text{(Eq 1)}$$

where σ_0 is the yield strength for material containing no grain boundaries and k_y is an experimentally determined coefficient. Other benefits of grain refinement are improvement in fracture resistance and superplastic behavior of steels with grain sizes <10 μm. The engineering design was mostly based on the ultimate tensile strength, which could be increased in normalized products by raising the carbon content to have more pearlite and by alloying, principally with manganese. Microalloying with Nb and V allowed normalized grain sizes as fine as 10 μm to be obtained in lower carbon, less-alloyed steels, with consequent improvement in yield strength, toughness, and weldability. This eventually led to a greater emphasis on yield, rather than tensile strength, in design. These increased interests in design have led to the ultra grain refinement of steels in recent years.

Hodgson et al. (Ref 1-3) first introduced the transformational grain refinement (TGR) processing steel strip to obtain a ferrite grain size of ~1 μm in a substantial fraction of its volume. This process was applied to thin strips, 2 mm thick, using hot reductions of approximately 40% to yield a product ~1.2 mm thick. In torsion simulations, it was found that dynamic recrystallization of ferrite in an interstitial-free steel, high-speed rolling could reduce the grain size very close to 1 μm, as a result of dynamic recrystallization (Ref 4). While rolling in the two-phase region, where dynamic recrystallization is also dominant, a grain size of 0.5 μm, leading to an average grain size of 1.5 μm was obtained (Ref 5). Recently (Ref 6), by varying the austenite grain size, the temperature of deformation, the rolling reduction, and the cooling rate after rolling, a grain size of 1 μm was obtained in the surface layers of a low-C, Nb-microalloyed steel. Priestner and Ibraheem (Ref 7) achieved grain sizes <1.5 μm through the thickness of a Nb-microalloyed steel rolled to approximately 2.5 mm using similar processing. They also proposed that the static recrystallization of hot band steel previously processed to a near-ultrafine grain size might be further refined by cold rolling and recrystallization.

In the present work, therefore, V-microalloyed steel has been used to investigate the recrystallization of ferrite cold rolled after initial TGR processing to near-ultrafine microstructure. Vanadium nitrocarbides are more soluble than niobium nitrocarbides at low austenitizing temperatures used in the TGR process and might be expected to precipitate during recrystallization after cold rolling. In addition, cold rolling might increase dispersion of fine cementite inherited from the TGR

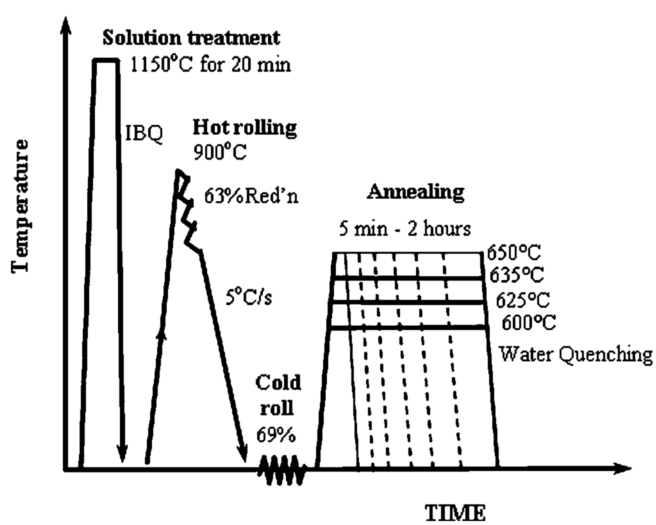

Fig. 1 Schematic representation of rolling and annealing schedule

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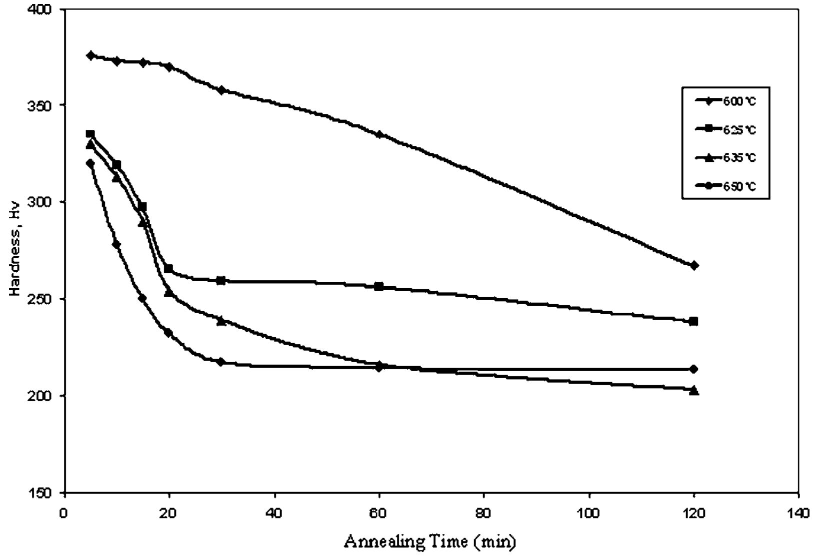

Fig. 2 Dependence of hardness on the annealing time and temperature

processing, to the point where it can be proposed to contribute by dispersion strengthening.

2. Experimental Procedures

The material used in the present studies was vanadium-microalloyed steel, provided in the form of hot-rolled slabs. The chemical composition of steels is as follows: C, 0.15; Si, 0.32; Mn, 1.4; P, 0.014; S, 0.009; Al, 0.04; N, 0.007; V, 0.43; and Fe, balance.

The optical metallographic studies revealed that initial microstructure of the steel was mainly ferrite and pearlite, having a minimum banded structure. To dissolve the alloying elements in solid solution, all the specimens from three steels were austinitized at 1150 °C, followed by iced brine quenching. Prior austenite grain size was measured by mean linear intercept technique. The specimens were hot rolled at 900 °C to reductions of 64% with the final thickness of 3 mm. The plates were then cold rolled to a reduction of about 70%, with a final thickness of ~1 mm. Small specimens of 6 × 15 mm were used from the sheet along the rolling directions, for annealing at different temperatures and times. The hot and cold rolling and annealing schedule is presented schematically in Fig. 1.

After undergoing the standard techniques of grinding and polishing, the specimens were etched in 2% nital solution and Marshel's reagent, to reveal the ferrite grain boundaries. Hardness was measured in Vickers scale, using 1 kg load. The electron backscattered diffraction (EBSD) technique was used for the measurements of high-angle grain boundaries (HAGB) and low-angle grain boundaries (LAGB) and their respective spacing.

The vanadium precipitation study was conducted on transmission electron microscopy (TEM), using thin-film carbon replicas taken on the copper mesh.

3. Results and Discussion

3.1 Static Recrystallization after Cold Rolling

The hot-rolling processes that have been used to achieve ultrarefinement of the ferrite grain structure depend on two basic grain refinement mechanisms. The first is TGR, which relies on the controlled application of deformation in the austenite phase field, to condition the austenite in such a manner that it transforms to a greatly refined ferrite microstructure during controlled cooling. The second is dynamic recrystallization of ferrite itself. In the present work, after austenitization at 1150 °C followed by quenching in iced brine solution, the grain size measured ~50 μm. The TGR process with a rolling reduction of 64% at 900 °C provided further grain refinement and average grain size measured 2.8 μm. The grain refinement by static recrystallization after cold rolling is a function of recrystallization temperature, degree of prior deformation, and grain size of prior deformation. Annealing after cold rolling, although it promoted the grain refinement by using the stored energy for recrystallization, has an adverse effect on hardness as a result of recovery. The annealing response to hardness after about 69% of cold rolling is presented in Fig. 2.

The hardness dropped smoothly but more rapidly at a higher temperature, due to enhancement in the recovery process. In a similar study on Nb microalloyed steels Priestner and Ibraheem (Ref 7) observed two plateaus of hardness drop. They claimed that hardness drop in the first plateau was not caused by recovery but instead was associated with abnormal grain growth. The second plateau was caused by the sudden release of stored energy, setting a different pattern of hardness drop. These plateaus are absent in the current study, and hardness dropped smoothly, which shows that V carbides precipitated in the early stage of annealing and pinned the recrystallized grains, thus retarding abnormal grain growth.

Figure 3 shows the TEM micrographs of V carbides taken on the thin film carbon replica, trapped on the copper mesh. The precipitates are mostly very fine but some coarsening was also observed. The results of the chemical analyses of these precipitates are presented in Fig. 4.

The peaks of copper are from the mesh used for catching the carbon replicas containing the precipitates. The major problem encountered in ultragrain refinement study was the poor toughness of the product observed by Japanese researchers in Super Metal Project, started in the late 1990s. A mixed microstructure with HAGB and LAGB, with minimum coarsening even after 2 h of annealing (as found in 4V steel) can rectify the problem

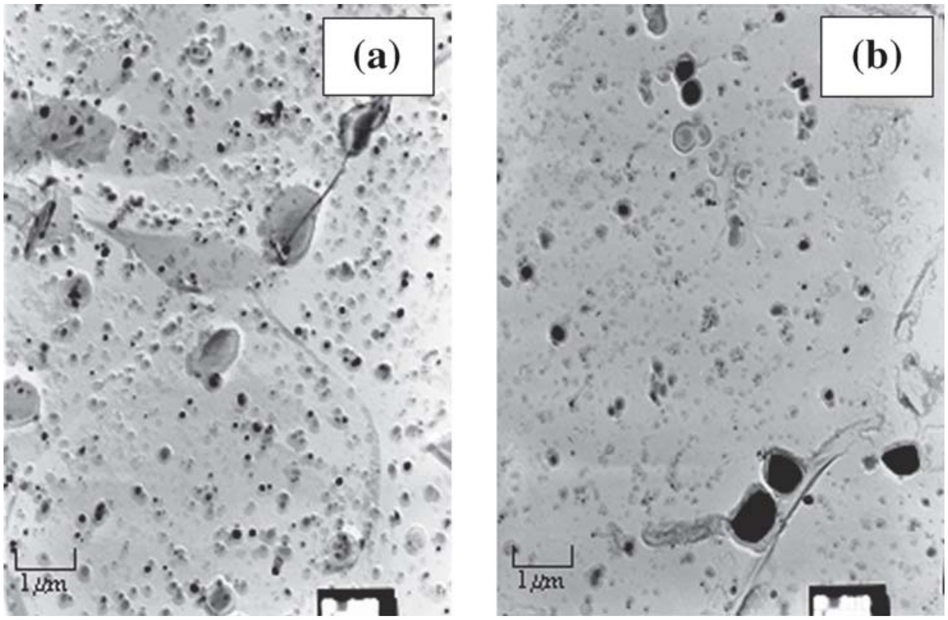

Fig. 3 TEM pictures showing V precipitates: (a) fine dispersion; (b) some coarser precipitate

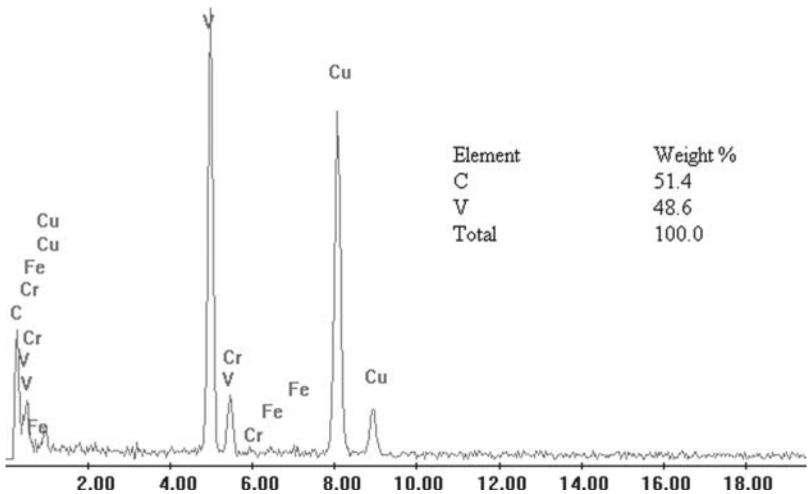

Fig. 4 TEM analysis of the V precipitates taken from extraction replica

because vanadium carbonitrites pinned the grain growth more effectively than Nb.

3.2 EBSD and Static Recrystallization

Figure 5 illustrates in a novel way the changes in the HAGB and LAGB or substructure during annealing where HAGB denotes misorientation >15°.

The annealing process started with similar LAGB and HAGB spacing, but the former increases with time were caused by movement of dislocations, resulting in the merging of LAGB. Similar trends were also observed at other annealing temperatures. The HAGB were not increased much by increasing the annealing temperature due to precipitation of V carbides and their pinning effect retarded the grain growth. In plain carbon steel, where precipitation hardening effect is minimum, HAGB should increase with LAGB spacing but HAGB remained almost constant in the present results during annealing, depicting minimum coarsening of grains. Priestner and Ibraheem (Ref 7) started the annealing process of Nb microalloyed steel at 600 °C with submicron grain size and after 60 min of annealing approximately 7 μm of grain size was observed, but in the current study, the HAGB spacing increased up to ~3 μm after 30 min of annealing at 650 °C. In the current study of V micoalloyed steel the process of annealing is much slower than Nb microalloyed steel. It is caused by the presence of LAGB or substructure, where even after 2 h of annealing, no abnormal grain growth was observed, as in the case of Nb-microalloyed steel.

4. Conclusions

Starting with the aim to study the grain refinement of V-microalloyed steel, by employing TGR and static recrystalli-

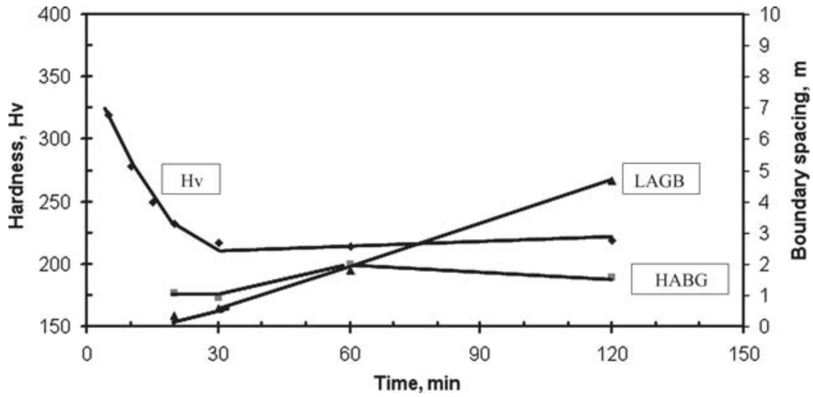

Fig. 5 Dependence of hardness and boundary spacing on annealing time at 600 °C

zation during annealing, the following conclusions were drawn:

- The TGR process refined the grain size to a reasonable extent, from 50 to 2.8 μm. The V precipitates dissolved in the earlier process by annealing at 1150 °C, would have precipitated out during heating to 900 °C and slowed down the recovery during hot rolling.
- During annealing of cold-rolled specimens, enough stored energy was provided for recrystallization, but the process of abnormal grain growth was sufficiently slowed down due to pinning of V precipitates. Therefore, V precipitates effectively controlled the grain size.
- The drop in hardness value during annealing was quite smooth, and no plateaus of hardness drop were observed, as quoted in the literature for Nb-microalloyed steel.

References

1. P.D. Hodgson, M.R. Hickson, and R.K. Gibbs, Ultrafine Ferrite in Low Carbon Steel, *Scripta Mater.,* 1999, **40,** p 1179-1184
2. P.J. Hurley and P.D. Hodgson, Formation of Ultrafine Ferrite in Hot Rolled Strip: Potential Mechanisms for Grain Refinement, *Mater. Sci. Eng. A,* 2001, **302,** p 206-214
3. P.J. Hurley, G.L. Kelly, and P.D. Hodgson, Ultra Ferrite Formation During Hot Strip Rolling, *Mater. Sci. Technol.,* 2000, **16,** p 1273-1276
4. A. Najafi-Zadeh, J.J. Jonas, and S. Yue, Effect of Dynamic Recrystallization of Grain Refinement of IF Steel, *Mater. Sci. Forum,* 1998, **113-115,** p 441-446
5. F.J. Humphreys, P.B. Prangnell, and R. Priestner, Fine-Grained Alloys by Thermomechanical Processing, *4th Int. Conf. on Recrystallisation and Related Phenomena,* T. Sakai and H.G. Suzuki, Ed., The Japan Institute of Metals, 1999, p 69-78
6. R. Priestner, Y.M. Al-Horr, and A.K. Ibraheem, Effect of Strain on Formation of Ultrafine Ferrite in Surface of Hot Rolled Microalloyed Steel, *Mater. Sci. Technol.,* 2002, **18,** p 973-980
7. R. Priestner and A.K. Ibraheem, Processing of Steel for Ultrafine Ferrite Grain Structure, *Mater. Sci. Technol.,* 2000, **16,** p 1267-1272