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Effect of Niobium on Austenite Recrystallization and Pearlite Colony Size in a Microalloyed Eutectoid Steel

H.-J. KESTENBACH and G. S. MARTINS

For many years, small additions of niobium have been used commercially to refine the ferrite grain size in hot-rolled products of low carbon steel. There are several ways in which niobium may lead to ferrite grain refinement.[1] The effect, however, is most pronounced when niobium is used to slow down recrystallization kinetics during controlled rolling.[2] In this case, plastic deformation during the final rolling passes is no longer accompanied by recrystallization. As a result, transformation to ferrite during cooling takes place in an elongated and highly dislocated austenite grain structure in which both a larger grain boundary area and a high stored energy of deformation increase the ferrite nucleation rate.

In order to retard dynamic or static recrystallization at finish rolling temperatures, niobium has to be taken into solution prior to rolling. This requirement poses no problem in low carbon steel where typical Nb additions in the range of 0.03 to 0.05 pct can readily be dissolved at ordinary soaking temperatures. For high carbon steel, however, the solubility of niobium in austenite is rather low. According to the solubility data of Nordberg and Aronsson,[3] only about 0.01 pct Nb can be dissolved at 1200 °C in a 0.8 pct carbon steel.

The solubility problem may explain why rather limited attention has been paid in the literature to niobium additions in high carbon steel. Such additions, however, appear to have been promising in development work for rail steels,[4] and have actually been used on a commercial scale in Brazil.[5] Furthermore, in a deliberate effort to extend microalloying techniques to higher carbon levels, Coladas *et al.*[6] have shown a strong delaying effect of 0.022 pct Nb dissolved in austenite on the static recrystallization of 0.4 pct carbon steel after hot rolling. The present note describes the effect of niobium on the dynamic recrystallization behavior during hot forging of a 0.75 pct carbon steel. A large decrease of the recrystallization volume fraction at deformation temperatures between 900 and 1050 °C has been related to the presence of only about 0.01 pct Nb dissolved in austenite prior to deformation.

Table I shows the chemical analysis of two experimental rail steels which were received in the as-rolled condition. Cylindrical specimens of 10 mm diameter and 20 mm height were cut from this material and subjected to single-pass deformation in the austenite range by drop forging. Prior to deformation, all samples were solution annealed at 1200 °C for 30 minutes in order to dissolve approximately 0.01 pct Nb[3] in the austenite of the niobium-bearing steel. This treatment led to average austenite grain diameters of 360 μm for steel NB-0 and of 220 μm for steel NB-1. Deformation temperatures were determined from standard cooling curves measured by inserting a thermocouple into test specimens cooled under identical conditions from the solutioning temperature. For each deformation temperature, true sample deformation was kept between 40 and 50 pct by adjusting the height of the falling weight. After deformation, the samples were transferred to a brine quenching bath in less than a second in order to preserve the as-deformed austenite grain structure. The austenite grain boundaries were revealed by etching in a hot saturated solution of picric acid in water after a tempering treatment at 510 °C for 16 hours.[7]

The effect of the niobium addition on the austenite recrystallization is shown in Figure 1 where the dynamically recrystallized volume fraction has been plotted as a function of deformation temperature for both the niobium-free and the niobium-bearing steel. The micrographs included in Figure 1 represent typical examples for the partially recrystallized austenite grain structure observed in central specimen regions after quenching. For steel NB-0, the first new grains started to appear at the old austenite grain boundaries after deformation at 800 °C. In the absence of niobium, recrystallization was virtually complete at 950 °C. In contrast, samples of the niobium-bearing steel deformed without any sign of recrystallization at temperatures up to 900 °C. Recrystallization did commence during deformation at 950 °C and reached a volume fraction of about 50 pct at the highest deformation temperature of 1050 °C.

In practice, the effect of niobium on the austenite recrystallization should lead to grain refinement after transformation, in order to improve steel properties. This cannot be accomplished by single-pass deformation because of the large austenite grain size which results from the solutioning treatment and which must first be refined by initial deformation passes at higher temperatures where niobium does not yet interfere with the occurrence of dynamical recrystallization. Some indication, however, of what could be the result of niobium grain refinement for the transformed

Table I. Steel Compositions in Weight Percent

Steel	C	Mn	Si	P	S	Nb
NB-0	0.76	0.89	0.16	0.014	0.021	—
NB-1	0.75	0.90	0.17	0.016	0.021	0.04

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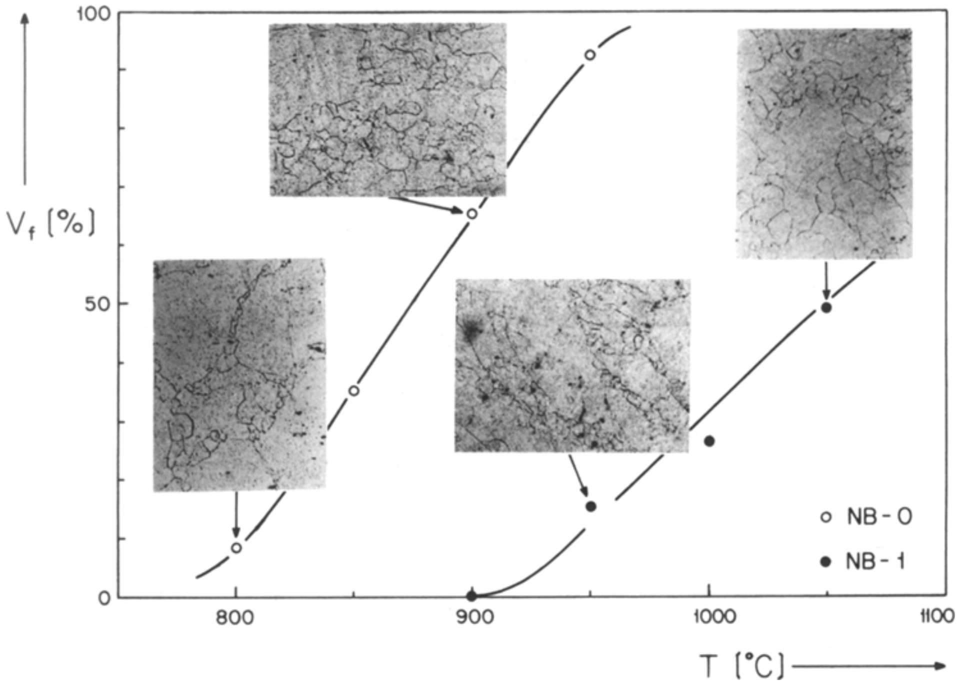

Fig. 1 — Effect of Nb on the recrystallized volume fraction, V_f, as a function of the deformation temperature, T. Micrographs show the partially recrystallized austenite grain structures quenched immediately after hot deformation. Magnification 40 times.

microstructure of a eutectoid steel is shown in Figure 2 which compares the austenite grain structure before transformation with the corresponding pearlite microstructure obtained when the brine quenching treatment was replaced by air cooling. In Figure 2(a), a rather coarse pearlite microstructure can be related to the large austenite grain size present after the solutioning treatment. Hot deformation at 900 °C has led to a much finer pearlite structure in both Figures 2(b) and 2(c). In Figure 2(b), dynamic recrystallization which occurred at 900 °C in steel NB-0 has mainly been responsible for the pearlite colony refinement. An austenite grain size of ASTM 5 (64 μm) was measured in completely recrystallized regions of this sample. In Figure 2(c), on the other hand, the austenite grain size has remained large (ASTM 1 or 250 μm approximately) because of the absence of recrystallization at 900 °C in steel NB-1. It can also be seen that a 50 pct deformation forging pass has not been very effective in elongating the austenite grains. Thus, the pearlite refinement in Figure 2(c) must have been caused primarily by the austenite deformation introduced before transformation.

The above results show that the small amount of niobium which does dissolve readily in the austenite of high carbon steel can have a rather strong effect on the austenite processing and the pearlite microstructure after transformation. It remains to be seen how this effect can be translated into useful mechanical properties. In the case of low carbon steel, both yield strength and toughness benefit directly from a ferrite grain refinement brought about by niobium additions. It is well known, however, that pearlite yield strength depends primarily on the interlamellar spacing. During continuous cooling, an enhanced pearlite nucleation rate may lead to larger interlamellar spacings because transformation would be completed at a higher temperature. Thus, the same effect of niobium on austenite recrystallization which was shown to refine the pearlite microstructure would tend to decrease yield strength. Fortunately, any niobium left in solution during and after the austenite deformation will show the opposite effect, *i.e.*, will lower the austenite decomposition temperature, thereby leading to smaller interlamellar spacings and a higher yield strength.

Another question to be answered concerns the mechanism by which a smaller colony size will change toughness. The simple brittle crack propagation concept which relates toughness to ferrite grain size in low carbon steel does not necessarily apply to the more complex pearlite microstructure. For example, toughness has been reported to depend on the prior austenite grain size rather than on the actual pearlite colony size.[8] Nevertheless, the possibility of using niobium for microstructure refinement in eutectoid steels should be pursued further.

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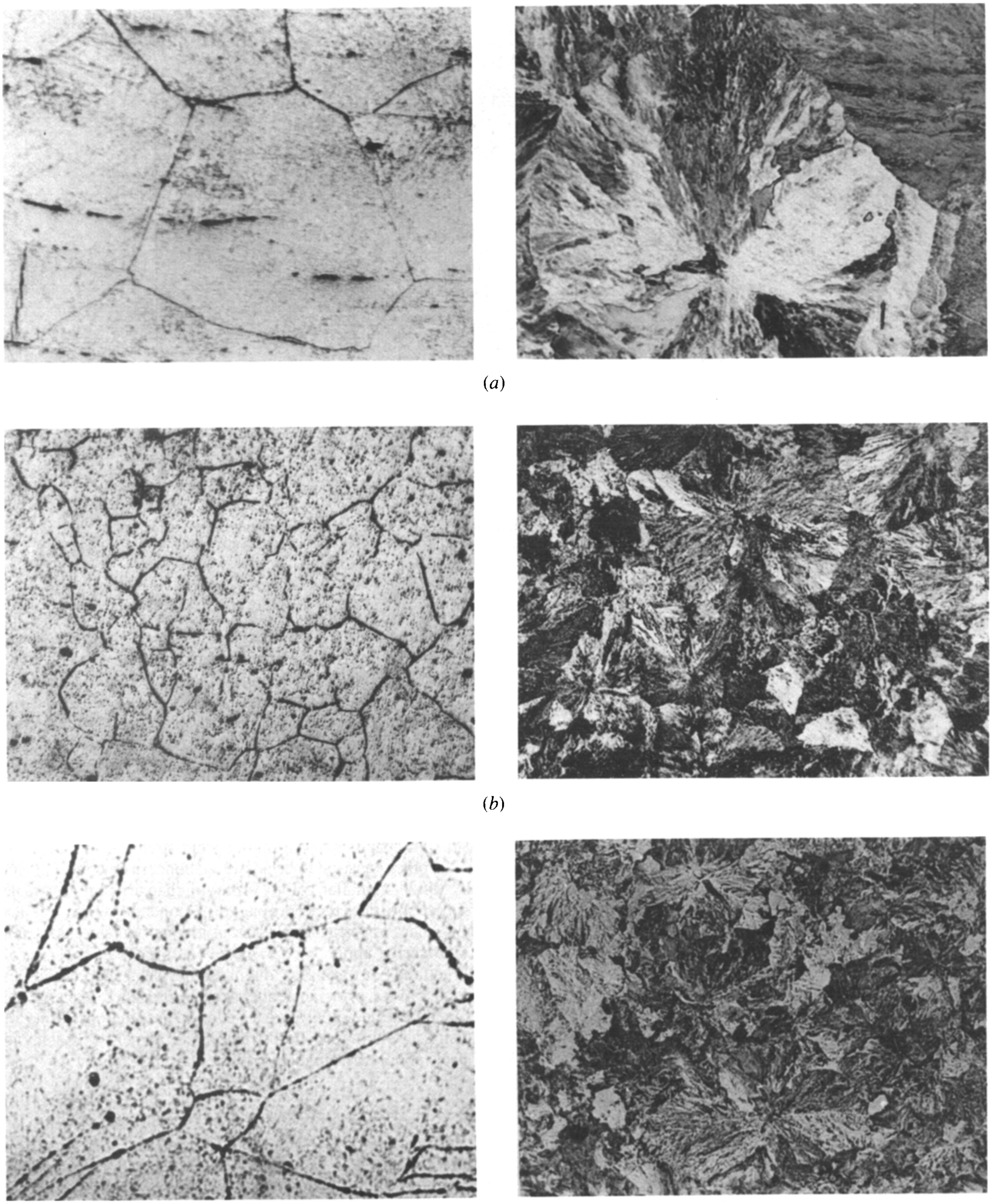

Fig. 2 — Effect of austenite grain structure on pearlite colony size after air cooling. Steel NB-0 solution-treated in (*a*). Steel NB-0 65 pct recrystallized during deformation at 900 °C in (*b*). Steel NB-1 unrecrystallized after deformation at 900 °C in (*c*). Magnification at left 150 times, at right 210 times.

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Segregation of Manganese during Intercritical Annealing of Dual Phase Steels

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As part of a study of the production of dual-phase steels by batch annealing, substitutional solute element partitioning of manganese during intercritical annealing was investigated. Such information is necessary to understand changes in volume fraction and hardenability of the austenitic phase and the mechanical properties of its transformation products produced during cooling.

In the batch annealing process, cooling is slow and high hardenability is required of the austenitic phase in order to obtain high-strength transformation products. Hence, high levels of alloy additions are required. Mn is the most economical alloying element which can provide this hardenability.

The kinetics of transformations during intercritical annealing have been studied both theoretically and experimentally by Wycliffe, Purdy, and Embury,[2,1] and experimentally by Speich, Demarest, and Miller.[3] Wycliffe *et al.* developed a detailed model based on local equilibrium, which enabled alloy element redistribution during annealing to be predicted. Evidence for Mn segregation was obtained in both above-mentioned studies. The purpose of the present communication is to provide further data on this phenomenon.

The chemical composition of the steel used was (in wt pct) 0.06 C, 2.83 Mn, 0.33 Si, 0.01 P, 0.01 S, and 0.03 Al. The steel was prepared as Al-killed induction melted 136 kg heats, and cast into rectangular ingots of 68 kg each. The ingots were split in half and soaked at 1200 °C for two hours and hot rolled to 25 mm thick slabs. Blanks (25 mm × 100 mm × 275 mm) were machined from these slabs, reheated at 1200 °C for 45 minutes, hot rolled to 3 mm thick plate, and immediately stored in a salt bath at 600 °C for one hour (to simulate coiling in a batch annealing production line), then cooled in air.

Small blanks were sheared from these plates and given the following intercritical annealing treatments:

(a) 690, 710, and 730 °C for 5 hours in Ar, brine quenched (BQ);
(b) 695 °C for 0.83, 5, 30, and 180 hours in Ar (30 hours and less) or vacuum (180 hours), brine quenched (BQ);
(c) 695 °C for 5 hours, furnace cooled (FC) at 28 °C/h to 250 °C, air cooled.

Metallographic samples for SEM examination were prepared after these treatments, and etched with saturated picral or stained with a 1:1 mixture of saturated picral and 1 pct $Na_2S_2O_5$ in distilled water. Thin foils for TEM were prepared from 1.5 mm diameter discs by electrolytic thinning.[4]

All the quenched steels had a dual-phase microstructure of martensite (α') grains in a ferrite (α) matrix as shown in Figure 1(a). The furnace cooled steel contained some pearlite (p) in addition (Figure 1(b)). Volume fractions f, measured from a series of micrographs taken from specimens representative of each heat treatment, are reported in Table I. Also reported in Table I are measurements of partition factors, to be described later.

The Mn distribution within the α' grains and the adjacent α matrix was determined in samples from the as-rolled steel and in samples given heat treatments (b) and (c). A 100 kV Philips 300 TEM equipped with an ORTEC energy dispersive X-ray spectrometer was used for the purpose. Typical results are shown in Figure 2 and a representative TEM micrograph in Figure 3. Mn levels were estimated from spectra obtained during 200-second counts, an example being shown in Figure 4. Fe and Mn intensities were measured by summation of the five highest counts in each peak, spanning approximately the upper half of the peaks. To estimate background, five points in an adjacent area of the spectrum on the low energy side of the Mn peak were used. The Mn:Fe ratio was calculated from the ratio of net intensities (after subtracting background) of the two peaks, neglecting atomic number corrections because Mn and Fe are adjacent in the periodic table. Three or four α' grains were examined for each heat treatment. The partition factor, defined as the ratio of Mn content averaged over the α' grain to Mn content

Table I. Volume Fractions* of Martensite and Pearlite, and Partition Factors

Heat Treatment	$f(\alpha')$, Pct	$f(p)$, Pct	$Mn(\alpha')/Mn(\alpha)$
as-rolled	13 ±2	0	1.10 ±0.02
690 °C, 5 h + BQ	13 ±1	0	
710 °C, 5 h + BQ	24 ±2	0	
730 °C, 5 h + BQ	34 ±3	0	
695 °C, 50 min + BQ	17 ±1	0	1.71 ±0.10
695 °C, 5 h + BQ	17 ±1	0	
695 °C, 30 h + BQ	17 ±1	0	2.30 ±0.05
695 °C, 180 h + BQ	18 ±2	0	2.61 ±0.14
695 °C, 5 h + FC	7 ±1	4 ±1	2.01 ±0.15

*Estimated from SEM micrographs by point counting according to ANSI/ASTM E 562-76

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