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# ON DISPERSION HARDENING OF MICROALLOYED HOT STRIP STEELS BY CARBONITRIDE PRECIPITATION IN AUSTENITE

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## 1. Introduction

Precipitation hardening by fine carbonitride particles has been used for many years in order to increase the strength of hot rolled microalloyed steels. During thermomechanical processing, carbonitrides may nucleate in austenite during rolling, at the  $\gamma/\alpha$  interface during transformation (interphase precipitation), or in supersaturated ferrite during final cooling [1]. It is generally believed, however, that a direct strengthening effect can only be obtained from partially coherent particles which have nucleated in ferrite during or after the phase transformation, and that prior precipitation in austenite, although important for grain refinement, will adversely affect the yield strength by reducing the amount of microalloy elements in solution which would otherwise be available for subsequent precipitation in ferrite [2–4]. On the other hand, extensive transmission electron microscopy observations of commercial hot strip steels conducted recently in the authors' laboratory suggested that fine carbonitride particles which nucleated during or immediately after rolling in unrecrystallized austenite may also cause precipitation strengthening [5,6].

In this paper, the authors try to re-examine the arguments which have been given previously in support of the exclusive role of ferrite-nucleated particles for precipitation strengthening, and to look for theoretical and experimental evidence which could support the idea of a significant contribution from carbonitride particles nucleated in austenite to the final strength of microalloyed hot strip steels.

# 2. Short Review of the Early Literature

When studying the literature about the early development of niobium microalloyed steels, it becomes apparent that the emphasis on strengthening by ferrite-nucleated particles can be traced back to just a few key observations:

-Early electron microscopy studies suggested that carbonitrides formed in ferrite were coherent with the matrix, because they could only be observed in thin film samples and not on extraction replicas [7]. In comparison, carbonitride particles could be extracted after a normalizing treatment which was known to reduce the yield strength of the as-rolled microalloyed steel [7,8]. Thus, during normalizing,

carbonitrides nucleated in ferrite would loose their strengthening potential on heating because of the loss of coherency during the  $\alpha \rightarrow \gamma$  phase transformation, just as carbonitrides nucleated in austenite would loose their strengthening potential on cooling because of the loss of coherency during the  $\gamma \rightarrow \alpha$  transformation. There are several arguments, however, which speak against such reasoning. According to theoretical calculations, coherency strains are not supposed to contribute significantly to dispersion strengthening when the Orowan mechanism is involved [9]. In addition, the large majority of thin film observations of carbonitride particles which were nucleated in ferrite do not exhibit the type of strain contrast which would be expected to come from coherent precipitates [10]. Finally, it was shown rather early that all that was needed to transfer the small ferrite-nucleated particles to replica samples was a lighter metallographic etch [11].

-A loss of precipitation strengthening was detected when precipitation in austenite was favoured by lower finish rolling temperatures [12] or by slower cooling rates during rolling [13]. In this case, however, thermomechanical processing conditions were such that the precipitation in austenite led to an average particle size of 20 to 30 nm [13], much larger than the carbonitride particles which nucleate on the deformation-induced dislocation substructure during finish rolling of commercial hot strip steels [5,6].

-In most of the early investigations on niobium microalloyed steels, carbonitride precipitation was studied in experimental steels where thermomechanical processing conditions were usually different from those encountered during industrial processing. In addition, many of the electron microscopy studies used extraction replicas in which it is difficult to distinguish between carbonitride nucleation in austenite or ferrite. Interestingly, in one particular investigation in which the particle origin could be identified, both precipitation in austenite as well as in ferrite were supposed to contribute to strengthening [11].

-The possibility of unambiguous identification of the origin of carbonitride particles in commercial microalloyed steels by electron diffraction has been known for a long time [14], based upon the determination of the particles' crystallographic orientation relationship with respect to the surrounding ferrite [15]. However, few authors have taken advantage of this technique in subsequent investigations.

#### 3. Precipitation Strengthening Model

The well-known Orowan-Ashby model, as presented recently by Gladman [16]:

$$\Delta\sigma = 10.8 \frac{\sqrt{f}}{d} \ln(1630d) \tag{1}$$

where  $\Delta\sigma$  represents the precipitation strengthening increment in MPa, f is the precipitate volume fraction and d the mean particle diameter in  $\mu$ m, has been used in Fig. 1 to evaluate the effects of the carbonitride particle size and volume fraction on the yield strength increment. This model has been shown previously to give very reasonable quantitative estimates for the precipitation strengthening potential of fine carbonitride particles in microalloyed steels [17,18]. It can be seen that, within the range of typical fine carbonitride distributions found in commercial microalloyed hot strip steels (particle diameters below 10 nm and volume fractions in the range of  $10^{-4}$  to  $10^{-3}$ ), the strengthening level depends strongly upon the volume fraction but is much less sensitive to particle size. It is this relatively small effect of particle size which suggests that a significant contribution to the strength of commercial hot rolled products can also come from carbonitride precipitation in austenite, as will be shown below.



Figure 1. Effects of the carbonitride volume fraction, f, and the particle diameter, d, on the precipitation strengthening increment,  $\Delta \sigma_{v}$ , according to the Orowan-Ashby model [16].

### 4. Example of Fine Carbonitride Precipitation in a Commercial Hot Strip Steel

During recent transmission electron microscopy observations of a commercial 0.12%C, 1.20%Mn, 0.33%Si, 0.06%Nb and 0.05%Ti hot strip steel of 7mm thickness and 534 MPa yield strength, duplex carbonitride distributions were encountered in several ferrite grains, indicating the presence of both carbonitride particles which had nucleated in austenite as well as somewhat finer particles which had nucleated during the  $\gamma \rightarrow \alpha$  transformation. Fig. 2(a) shows the general ferrite-pearlite microstructure of the as-coiled material, while Fig. 2(b) shows one of those ferrite grains in which carbonitride precipitation was analyzed by transmission electron microscopy and diffraction. Particle size and volume fractions were determined in three separate areas denominated "I" (for interphase precipitation), "A" (for austenite precipitation) and "G" (for general precipitation) in Fig. 2(b). Portions of two of these areas are shown at a higher magnification in Fig. 3. In Fig. 3(a), precipitate row formation close to the



Figure 2. Microstructure of a commercial 0.06%Nb/0.05%Ti microalloyed hot strip steel. Optical microscopy showing ferritepearlite structure in (a). Transmission electron microscopy showing ferrite grain with austenite + interphase precipitation in (b). Local precipitation strengthening potentials were analyzed in areas I, G and A according to the Orowan-Ashby model. See text for discussion.



Figure 3. Details of carbonitride precipitation in ferrite grain shown in Fig. 2. Interphase precipitation rows close to the grain boundary and general carbonitride precipitation in (a). Bright field of general carbonitride precipitation in (b), dark field of same area with carbonitride precipitation in austenite only in (c). Diffraction pattern showing a <110> ferrite orientation and a  $\{200\}$  carbonitride dark field reflection in (d).

grain boundary allowed at least some of the finer particles to be identified with interphase precipitation, and only these row particles were counted for the Orowan-Ashby analysis of region "I." With respect to area "A," both smaller and larger particles were found under bright field conditions in Fig. 3(b), while many of the larger particles but none of the smaller ones in this area were illuminated under dark field conditions in Fig. 3(c). The crystallographic orientation of these larger particles was determined from the electron diffraction pattern shown in Fig. 3(d) where the position of the objective aperture identifies the 200 carbonitride reflection which was used to illuminate the particles in Fig. 3(c). This position does not obey the Baker-Nutting relationship [19] which proves that most of the larger carbonitride particles shown under bright field conditions in Fig. 3(b), and all the particles shown under dark field conditions in Fig. 3(c), must have nucleated in austenite. For the Orowan-Ashby analysis of region "A," particle size and volume fraction were determined for the dark field conditions shown in Fig. 3(c). Finally, both smaller and larger particles were measured and counted under bright field conditions in area "G" which was supposed to include both austenite and interphase precipitation.

In order to determine volume fractions, the sample thickness was measured by counting the number of thickness fringes produced under controlled two-beam contrast conditions [20] in both grain boundaries which can be recognized in Fig. 2(b), indicating that the sample thickness increased from

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Precipitation Orgin	Sample Area	Sample Thickness [nm]	Mean Particle Diameter [nm]	Particle Volume Fraction	$\Delta\sigma$ [MPa]
Austenite	А	100	4.5	$1.85 \times 10^{-4}$	65
Interphase	Ι	117	2.0	$1.90 \times 10^{-4}$	85
General	G	111	2.2	$3.15 \times 10^{-4}$	112

TABLE 1 Quantitative Estimates of Precipitation Strengthening

about 88 nm at the bottom right-hand corner to about 117 nm at the top left-hand corner of the micrograph.

The principal data of this analysis have been assembled in Table 1 which, as the most important result, shows rather similar yield strength increments associated with austenite and interphase precipitation (65 and 85 MPa, respectively).

#### 5. Discussion

It is commonly believed that the particular processing conditions of hot strip rolling which include very short interpass times during finish rolling and rapid cooling on the run-out table should severely limit the extent of carbonitride precipitation in austenite. It should be recognized, however, that the volume fraction of austenite precipitation will continue to increase through particle growth after the  $\gamma \rightarrow \alpha$  transformation. This means that, during coiling, both interphase and austenite-nucleated particles will compete for microalloy atoms which have remained in solution, a process which, possibly, could lead to similar volume fractions as shown in Table 1.

It is also generally accepted that the level of dispersion strengthening by the Orowan mechanism is inversely proportional to the distance between particles. As judged qualitatively from simple electron microscopy observations, Fig. 3, this distance appears to be much larger in the case of austenite precipitation, Fig. 3(c), when compared to a typical example of fine interphase precipitation, Fig. 3(a). However, it is important to remember that the effective interparticle distance, L, must be measured on the slip plane and, in general, will be much larger than the apparent interparticle distances which appear on a projected transmission electron micrograph [16]. Thus, only a small number of the particles which are present in Fig. 3 would be intercepted by any given slip plane. Although, for a given volume fraction, L increases rapidly with the mean particle size, the probability that a particular carbonitride particle will be intercepted by any given slip plane (and thus contribute to strengthening) will also be larger when the particle size increases. This is the reason for the rather small effect of particle size on precipitation strengthening as shown in Fig. 1 and, at the same time, explains why both the finer interphase precipitation in Fig. 3(a) and the coarser austenite precipitation in Fig. 3(c) show rather similar dispersion strengthening potentials.

#### 6. Conclusions

Theoretical arguments as well as experimental evidence from an industrially processed Nb-Ti microalloyed steel have been presented to show that carbonitride particles nucleated in unrecrystallized austenite may contribute significantly to the final strength of commercial hot strip steels.

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