

# Experimental evaluation of the particle size dependence of the dislocation–particle interaction force in TiC-precipitation-strengthened steel

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The particle size dependence of the interaction force per TiC particle was experimentally evaluated from the amount of particle strengthening and the particle spacing. The amount of pure particle strengthening by TiC precipitates was successfully estimated from ferritic model steels with significantly low dislocation density and homogeneously dispersed fine precipitates. The interaction force indicated that very fine TiC particles are effective and that an equivalent volume diameter of 2–3 nm is the most potent for particle strengthening.

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Carbide precipitates, such as titanium carbide (TiC), niobium carbide (NbC) and vanadium carbide (VC), have been used for particle strengthening in commercial steel. It is reported that nanometer-size carbide or carbonitride precipitates create a high degree of particle strengthening in ferrite [1–3]. Practical high-strength low-alloy (HSLA) steels gain strength from not only particle strengthening but also from grain size refinement, dislocation density and solid solution strengthening. The contribution of particle strengthening in Nb/Ti-added HSLA steel was estimated by assuming the addition law of strengthening from dislocations and particles [1]. In a Nb-microalloyed steel, it has been suggested that Nb-rich Guinier–Preston-zone-like clusters provide much more strengthening than subsequent Nb(C,N) precipitates with aging, although the Cottrell atmosphere and the recovery of the microstructure also change simultaneously [3]. Although determining the magnitude of particle strengthening from such precipitates or clusters has been attempted, there is still difficulty in separating strengthening from other sources. In addition, heterogeneous nucleation occurs in those steels that have found practical applications, which makes it difficult to evaluate accurate

particle spacing, an important parameter with respect to classical particle strengthening theories.

According to classical theories, the amount of particle strengthening depends on the interaction (resistance) force per particle with dislocation and the spatial distribution of the particles [4]. Thus, for meaningful materials design, it is important to understand the variation in the interaction force with respect to the precipitation species and particle size. Takaki et al. predicted the critical shearable/nonshearable particle diameter based on the hardness of various carbides, assuming the frictional force effect to be the particle-strengthening mechanism; the diameter for TiC is 7 nm, which is the hardest of the carbides [5].

To evaluate the capacity for strengthening purely from particles and its size dependence, we conducted experiments that achieved both of the following two aspects: to eliminate strengthening other than particle strengthening, and to nucleate precipitates homogeneously. To meet the objective, in this study we adopted a model steel without a  $\gamma \rightarrow \alpha$  phase transformation and estimated the amount of pure particle strengthening due to TiC particles in isothermally aged steel with a sufficiently low and constant dislocation density. After correcting for the effect of the plate-like particle shape on particle spacing, the particle size dependence of the interaction force per TiC particle was estimated according to the same approach as applied to shearable Cu

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particles in steel by Takahashi et al. [6]. For a very small TiC precipitate, it is considered that the possibility of shearable particles should be taken into account. Since some degree of error is unavoidable when determining absolute values of the interaction force, it makes sense to discuss the relative values.

A ferritic model steel with a chemical composition of Fe–0.03C–0.10Ti–0.2Mn–3.0Al (wt.%) was hot rolled to a thickness of 12 mm. Round bars (diameter, 11 mm; length, 95 mm) were cut from the hot-rolled plate and annealed at 1300 °C for 10 min, then quenched into stirred water for solution treatment. By adding a sufficient amount of Al, the model steel used in this study remained ferritic throughout the solution treatment. After the treatment, it was verified by the electrolytic extraction method that there was barely any residual TiC precipitate. The quenched sample bars were aged at 580 °C for 0.25–512 h (listed in Table 1) for the precipitation of TiC. Aging at 400 °C for 24 h and subsequently at 200 °C for 4 h was performed on another sample bar to reduce the dissolved C sufficiently for the precipitation of coarse cementite. Hereafter, the sample with cementite is denoted as the low-temperature-aged sample. The optical microstructure of the samples was polygonal ferrite with large grain size of approximately 1 mm. Transmission electron microscopy (TEM) observations showed that the dislocation density of the samples was as sufficiently low at approximately  $10^{12} \text{ m}^{-2}$  as that of the well-annealed ferrite. These microstructures did not change with aging time, which indicates that the strengthening due to dislocation and grain size was unchanged throughout aging.

Tensile tests were performed to measure the 0.2% proof stress as a representation of the yield strength of each sample. Tensile test specimens with a gage length of 24 mm and a diameter of 6 mm were taken from the cores of aged bars and an unaged (as-quenched) bar to avoid the decarburized region. The test speed was set at  $15 \text{ MPa s}^{-1}$ .

The 0.2% proof stress value of the aged samples changed dramatically with holding time at 580 °C, as shown in the first line of Table 1. An under-aging region was observed from 0 to 4 h, a peak-aging region was seen from 8 to 32 h, and an over-aging region was detected from 64 to 512 h. The proof stress of the low-temperature-aged sample was nearly 100 MPa lower than the unaged sample, which was considered to be the loss of solid solution strengthening due to C, because neither of the samples contained any fine precipitate that would be effective for particle strengthening. The differential of the proof stress between the 580 °C aged and the low-temperature-aged sample is regarded as the particle strengthening. However, the solid solution strengthening due to C also must be deducted from the sample in the initial stage of aging, which contains dissolved C.

The concentration of dissolved C in each sample was measured by atom probe tomography (APT) analysis. The same crystal orientation was carefully chosen for

the analysis, and the concentrations were compared relatively, because there is an anomalous enrichment of dissolved C in specific crystal orientations during the APT analysis process [7]. Based on the differences in the proof stress and the concentration of dissolved C between the unaged and the low-temperature-aged samples, the solid solution strengthening per 1% of dissolved C was estimated. It is assumed that solid solution strengthening is proportional to the concentration of the solute atoms, based on a number of experimental results [8]. By this means, the amount of solid solution strengthening due to C was calculated from the concentration of dissolved C in each sample, as determined by APT. In the second and third lines of Table 1, estimated solid solution strengthening and particle strengthening of each sample are listed. The particle strengthening was as high as 320 MPa in the peak-aging region, which indicates that high-number-density precipitation occurs homogeneously. It is noted that the estimated particle strengthening in the 0.25 h aged sample is too small and uncertain, and thus the interaction force in the sample could not be evaluated.

APT has the advantage of offering a thorough observation of solute atoms and fine clusters and precipitates that are subnanometer to nanometers in size. Measurement of the concentration of dissolved Ti solute atoms aids the accurate estimation of the volume fraction of precipitation. The APT analyses were conducted with the same instrumentation and software as in the previous study [6], at a specimen temperature of 65 K, a pulse fraction of 20–25%, and a pulse frequency of 20 kHz. A peak at a mass-to-charge ratio of 24 was assigned as  $\text{Ti}^{2+}$  in this study. Although the peak at 24 also comprises  $\text{C}^{2+}$ , it is expected to be minor. The cluster analysis (particle analysis) was performed by the maximum separation method [9]. The maximum separation distance ( $d$ ) and the minimum number of solute atoms ( $N_m$ ) were assumed to be 1.0 nm and 10, respectively.

Figure 1 shows the three-dimensional (3-D) Ti maps for the unaged and aged samples at 580 °C taken by APT analysis. Only Ti atoms are displayed for visibility. While the Ti atoms in the unaged sample are randomly dissolved, very small TiC clusters with indeterminate form appear in the 0.25–1 h aged samples. In the samples aged for 2 h and longer, plate-like TiC precipitates are observed. The TiC clusters and precipitates a few nanometers in size and with high number density are uniformly distributed, indicating that the TiC precipitates nucleated homogeneously in the matrix. The size of the precipitates increases with aging time. While the size distribution of the TiC precipitates is not wide until the beginning of the peak-aging region, it begins to widen from the latter part of the peak-aging region. The amount of dissolved Ti atoms seems to decrease in the under-aging region.

Figure 2 shows the evolution of (a) the diameter and (b) the number density of the TiC precipitation particles

**Table 1.** The 0.2% proof stress, solid solution strengthening by carbon, and estimated particle strengthening as a function of aging time at 580 °C.

Aging time at 580 °C (h)	0	0.25	0.5	1	2	4	8	16	32	64	128	256	512	400 °C 24 h (base)
0.2% Proof stress (MPa)	345	334	361	370	468	523	573	568	564	541	509	452	420	247
Solid solution strengthening by C (MPa)	98	100	50	64	38	–	–	–	–	–	–	–	–	–
Particle strengthening (MPa)	0	0	65	59	183	276	326	321	317	294	262	205	173	0

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