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Original Article

Effect of vanadium and niobium alloying elements on the microstructure and mechanical properties of a drop forging microalloyed HSLA steel

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Abstract

This research explains the results of microstructure and mechanical property investigations of four drop forging microalloyed HSLA steels, ME Wrench 1, ME Wrench 2, ME Wrench 3, and ME Wrench 4. The purpose was to find the influence of vanadium (0.121, 0.119, 0.120, and 0.210%) and niobium (0.00045, 0.00055, 0.00052, and 0.00057%) content on the microstructures of ME Wrench 1 in comparison to ME Wrench 2, 3, and 4. It has been found that the group of ME Wrench 1 shows most evenly distributed proeutectoid ferrite as highest volume fraction and finest prior austenite grain size in theirs structure. At the highest vanadium intensity and nitrogen appearance of ME Wrench 1 V(CN) precipitation dispersion occurs in non-random way. Effective yield stresses have been controlled by applying different mean ferrite grain size according to Hall-Petch equation. It has also been noted that grain size and yield stress are in good correlation.

Keywords: vanadium and niobium alloying elements, microstructures, mechanical properties, drop forging, microalloyed HSLA steel

1. Introduction

The name “microalloyed steels” was first applied to a class of higher strength low carbon steels containing small additions of niobium and/or vanadium. Any attempt at a rational definition of microalloying based on the increases in strength produced by small additions would now include aluminium, vanadium, titanium and niobium-treated steels. Such steels contain essentially less than 0.1% of the alloying additions used singly or in combination; by these yield strength increments of two or three times that of plain carbon-manganese steel can be attained (Gladman, 1977). Microalloying and controlled processing is a well-known, direct, and cost saving way to increase the strength level for ferrite/pearlite structures, successfully adopted for HSLA steels (Holappa *et al.,* 2001). Ultrahigh strength low carbon microalloyed steels (Ti, Nb, V) have been developed for line pipe application considering the extreme conditions prevailed across the globe (Pattanayak *et al.,* 2015). The properties considered here are strength, which is fundamental to the concept of microalloyed steel, and the ancillary, but nonetheless highly important, properties of toughness, ductility, and formability. These properties depend upon microstructural features in different ways and in some other cases depend upon different microstructural features. Most strengthening mechanisms have a greater effect on the yield strength in terms of both absolute and relative magnitudes. The weight saving achieved through the use of microalloyed steels would certainly have been reduced considerably had design stresses remained linked to the tensile strength (Gladman, 1977). In alloy design, various relationships between structure and properties are established to guide the selection of composition and microstructure such as yield strength and hardness are measured to correlate with the composition and microstructure (Tien and Ansell, 1976).

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The four drop forging microalloyed HSLA steels with the wrench trade name of ME Wrench 1, ME Wrench 2, ME Wrench 3, and ME Wrench 4 are shown in Figure 1 that have engaged in supplying professional tools for more than 30 years. Precision forging technology in forging process is shown in Figure 2, where metal is moved while still in the solid state. The four drop forging are specialized in forging technology to create better metal configuration. Non-straight grain flow and non-sharp corners can extend the lifetime and durability of sockets. Torque is transmitted for powerful tightening under safety. The four drop forging tools control heat treatment system to ensure the right hardness without brittleness. From microstructure of these tools, the elements are well-distributed as in Figure 3 (Fan *et al.*, 2014; King Tony, 2015).

1.1 Grain refinement as a strengthening mechanism

It was well known that aluminum treated steels have higher yield strengths and superior toughness, and would have a much finer ferrite grain size, and these steels were produced as bulk tonnage steels. The quantitative relationship between yield strength and grain size in metals was first described by Hall, based on experimental observations, and by Petch, based on both experimental and theoretical approaches (see Askeland, 1996). These workers had collaborated and the experimental evidence was based on observations in steel. The relationship between yield strength and grain size is now commonly known as the Hall-Petch equation.

$$\sigma_y = \sigma_i + k_y d^{-1/2} \tag{1}$$

where σ_y is the yield strength or the stress at which the steel permanently deforms or as the lower yield stress, σ_i is the constant or friction stress, k is the strengthening coefficient and d is the average diameter of the ferrite grains (Gladman, 1977). This type of relationship holds for a wide variety of irons and steels as well as for many non-ferrous metals and alloys (Honeycombe, 1982).

Ferrite grain size can be measured by several different methods (Kurzydłowski and Ralph, 1995). One of the earliest methods of evaluating of microalloyed steels was developed by Spektor. He considered the penetration of a polydispersed system of spheres by a straight line or secant. There are many spheres of diameter d_j, the centers of which are distributed in space with statistical uniformity. The distance from the center of the sphere to the intersecting chord is:

$$x(i,j) = \left[\left(\frac{d_j}{2}\right)^2 - \left(\frac{l_i}{2}\right)\right]^{0.5} \tag{2}$$

where l_i is the length of the chord. The number of chords per unit length of the secant having lengths between i_j and d_j is calculated next. There are $N_v(j)$ centers of spheres per unit volume of the structure, the number of chords per unit length of the secant will be:

Figure 1. Same four drop forging microalloyed HSLA steels as the name of ME Wrench 1, ME Wrench 2, ME Wrench 3, and ME Wrench 4 are open end wrenches, the difference of these four wrenches are the content of wt%, open end angle, size in mm, and weight in gramme.

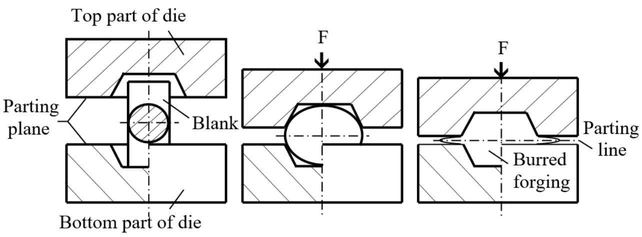

Figure 2. Single-impression closed-die drop forging process of the same four open end wrenches microalloyed HSLA steels is also produced the tools of ME Wrench 1, ME Wrench 2, ME Wrench 3, and ME Wrench 4 (Vladimirov, 1977).

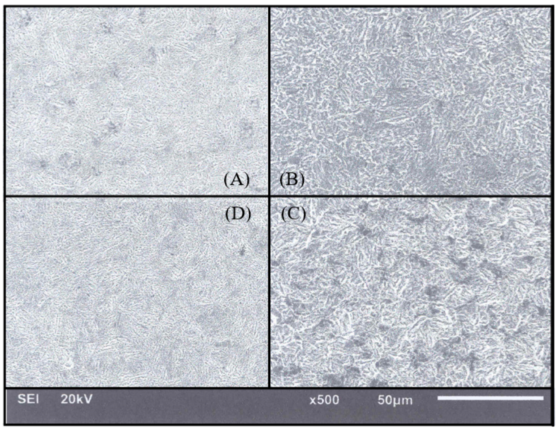

Figure 3. Microstructures of the four drop forging tools, ME Wrench 1 number 4) from Table 3 as (A) in this figure, is the highest yield strength of all steels, ME Wrench 2 number 4) from Table 3 as (B) in this figure, is the highest yield strength of this group steels, ME Wrench 3 number 3) from Table 3 as (C) in this figure, is the highest yield strength of this group steels, and ME Wrench 4 number 3) from Table 3 as (D) in this figure, is the highest yield strength of this group steels, They have been controlled heat treatment system to ensure the right hardness without brittleness, all photographs have same magnification (Fan *et al*, 2014).

$$N_L(i,j) = \pi x^2(i,j) N_v(j) = \frac{\pi}{4}\left(d_j^2 - l_i^2\right) N_v(j) \tag{3}$$

It is sufficient to represent the distribution of particle size as a discontinuous function with a limited number of class intervals. The working formula is obtained (Ganwarich, 2005)

$$N_v(j) = \frac{4}{\pi\Delta^2}\left[\frac{n_L(j)}{2j-1} - \frac{n_L(j+1)}{2j+1}\right] \quad (4)$$

1.2 Forming of structural components

Formability as a forging is a manufacturing method in which compressive forces are used to shape a metal slug into a near-net shape product with the use of a tool. The method is adaptable for a range of shapes, from simple to the very complex; for intricate geometries, forging is performed using several tool sets as multi-step process as shown in Figure 2. Forgings are typically classified according to the type of tooling used in the forging process; these include drop forging and impression-die forging (Mallick, 2010). Drop forging is a forging process where a hammer is raised and then "dropped" onto the work piece to deform it according to the shape of the die. There are two types of drop forging: *open-die drop forging* and *closed-die drop forging*. As the names imply, the difference is in the shape of the die, with the former not fully enclosing the work piece, while the latter does. Precision forged parts have good fatigue life and good intergranular corrosion resistance; this is a result of the process creating improved grain alignment within the part (Wikipedia, 2015).

2. Experimental Procedures

Four wrenches have been forged by the process design for Kittikun Motor's factory at Bangbon, Bangkok, Thailand, 10600, and have been adjusted already for the ME Shop, Srinakharinwirot University, Thailand. The test has been performed by the group of four grades as open end wrenches of drop forging microalloyed HSLA steels are as follows, 1) ME Wrench 1, 2) ME Wrench 2, 3) ME Wrench 3, and 4) ME Wrench 4.

The group of four samples has been studied to optimize the influence of carbon content and single and multiple microalloying additions on the austenite and ferrite grains size microstructure as-forging state in Figure 2 and as-annealed of various temperatures after forging state. The composition (wt%) of these steels used in this study is given in Table 1 and 2. All steels are microalloyed HSLA with vanadium, titanium, niobium, and also aluminium, which is predominantly used for precipitation hardening of ferrite. ME Wrench 1 is a microalloyed HSLA steel for the comparison with other steels of number 2-4 and is additionally microalloyed with titanium making use of small TiN-particles for effective inhibition of austenite grain growth during reheating and hot rolling before

Table 1. Chemical composition of the four drop forging tools microalloyed HSLA steels as ME Wrench 1, ME Wrench 2, ME Wrench 3, and ME Wrench 4, wt %.

Steels/ Elements	ME wrench 1	ME wrench 2	ME wrench 3	ME wrench 4
C	0.427	0.434	0.335	0.400
Si	0.247	0.221	0.299	0.229
Mn	0.767	0.758	0.662	0.764
P	0.0219	0.0155	0.0112	0.0179
S	0.0104	0.0055	0.0038	0.0074
Cr	0.539	0.525	0.620	0.541
Mo	0.00044	0.0011	0.0377	0.0104
Ni	0.0151	0.0824	0.155	0.0390
Al	0.0469	0.0369	0.0299	0.0251
Co	0.0042	0.0052	0.0112	0.0070
Cu	0.0101	0.0205	0.219	0.103
Nb	0.00045	<0.00020	0.00033	0.00026
Ti	0.0039	0.0018	0.0022	0.0034
V	0.121	0.116	0.0862	0.104
W	0.0047	0.0042	0.0066	0.0057
Pb	0.0020	0.0023	0.0020	0.0018
Sn	0.00040	0.00061	0.0121	0.0046
B	0.00025	0.00023	0.00036	0.00027
N	0.0031	0.0017	0.0113	0.0093
Fe	97.7	97.7	97.7	97.6

Table 2. Vanadium and Niobium Alloying Elements in the four drop forging microalloyed HSLA steels as ME Wrench 1, ME Wrench 2, ME Wrench 3, and ME Wrench 4; assumption with other alloying elements from Table 1 are constant, wt %.

Steels/ Elements	ME wrench 1	ME wrench 2	ME wrench 3	ME wrench 4
V	1) 0.121	1) 0.116	1) 0.0862	1) 0.104
	2) 0.119	2) 0.113	2) 0.0866	2) 0.104
	3) 0.120	3) 0.113	3) 0.0861	3) 0.104
	4) 0.210	4) 0.114	4) 0.0863	4) 0.104
Nb	1) 0.00045	1) <0.00020	1) 0.00033	1) 0.00026
	2) 0.00055	2) 0.00037	2) 0.00036	2) 0.00036
	3) 0.00052	3) 0.00037	3) 0.00026	3) <0.00020
	4) 0.00057	4) 0.00031	4) 0.00032	4) 0.00027

drop forging. Furthermore, three microalloying variants ME Wrench 2, ME Wrench 3, and ME Wrench 4 were prepared as shown in Table 1. ME Wrench 2, ME Wrench 3, and ME Wrench 4 have enhanced contents of vanadium and steel ME Wrench 3 is microalloyed with high niobium to elevate the recrystallization temperature of austenite. The evaluation of microstructure during the process of a single-impression closed-die drop forging and of annealing were analyzed by methods of qualitative and quantitative metallography by Spektor analysis theory and Hall-Petch relationship (measurement of the chord lengths of austenite grain size distribution and determination of the yield stress of polycrystalline aggregate in which ferrite grain size is the only variable. The working fomular is

$$G = M + \left(6.64 \log \quad \frac{g}{100} \right) \tag{5}$$

where G is the index number of the International Standard (ISO643), M is the number of the closest standard chart, modified as a function of the ratio of the magnifications, g is the magnification of the image on the screen or photomicrograph is not x 100, $(N_v)_j$ is the number of particles of mean diameter per unit volume in the interval of j, $(n_L)_j$ is the number of chords per unit length of test line, σ_y is the yield strength or the stress at which the material permanently deforms, σ_o is a constant stress, K is a material constant and d is the average diameter of the ferrite grains.

A microscope was used to investigate details of the microstructure (morphology of ferrite, pearlite, nonmetallic inclusions, micro-cracks, and also precipitates) at magnifications from x75 to x1,500. Microstructures are an indication of the quality of heat treatment and of mechanical properties. Representative photomicrographs were taken of the grain size microstructures of the entire relevant samples. In order to determine the grain size, the specimens were specially polished and etched in a saturated piric acid solution at about 80°C and a 2% nital solution (Lakhtin, 1977) of austenite and ferrite microstructure, respectively. The concentration of the acid had to be adjusted for different samples. The grain size was then measured at x75 using a filler eyepiece. With respect to macro- and microhardness measurements were carried out in order to characterize the global hardness of the complex microstructure as well as the hardness of its constituents (Ganwarich, 2003, 2005).

3. Results and Discussion

Figure 3 shows microstructure of the four drop forging tools as ME Wrench 1, ME Wrench 2, ME Wrench 3, and ME Wrench 4. Table 3 shows size distribution and yield strength of polygonal-acicular ferrite grains of the four group drop forging microalloyed HSLA steels. The variation of yield strength with $d^{-0.5}(m^{-0.5})$ is shown in Figure 4. The best microalloyed steel is ME Wrench 1, shown in row 4, Table 3.

Table 3. Size distribution and yield strength of polygonal-acicular ferrite grains of the four groups drop forging microalloyed HSLA steels from the Table 2, the ME Wrench 1 shows highest yield strength, so, in this steel of number 4 that has highest V and Nb alloying elements, and other group shown that ME Wrench 2, ME Wrench 3, and ME Wrench 4.

1 Steels	2 Range of chord lengths, µm	3 Number of chordsper mm., $(n_L)_j$	4 Diameter of grains, mm,d_j	5 Number of grain per mm^3,$(N_v)_j$	6 Evaluated mean grain size, µm, d	7 $\sigma_y = \sigma_o + Kd^{-0.5}$ (MN.m^{-2})
ME Wrench 1	1) 0-520	366	0.052-0.520	2.0260×10^5	**19.95**	**314.26**
	2) 0-300	1376	0.030-0.300	26.8228×10^5	**13.30**	**404.42**
	3) 0-260	1250	0.026-0.260	28.1772×10^5	**10.10**	**425.47**
	4) 0-280	1752	0.028-0.280	45.3428×10^5	**09.60**	**435.37**
ME Wrench 2	1) 0-494	348	0.049-0.490	1.9247×10^5	21.01	307.32
	2) 0-293	1342	0.029-0.293	26.15223×10^5	3.50	373.40
	3) 0-247	1219	0.024-0.247	26.76834×10^5	10.63	415.72
	4) 0-266	1665	0.026-0.266	43.07566×10^5	10.11	425.36
ME Wrench 3	1) 0-500	360	0.050-0.500	2.1256×10^5	20.30	311.87
	2) 0-280	1300	0.028-0.280	25.48166×10^5	13.59	372.29
	3) 0-255	1250	0.025-0.255	27.05011×10^5	11.63	399.21
	4) 0-270	1549	0.027-0.270	40.80852×10^5	12.14	391.58
ME Wrench 4	1) 0-480	375	0.048-0.480	2.0089×10^5	20.65	309.57
	2) 0-277	1380	0.027-0.277	24.14052×10^5	14.10	366.23
	3) 0-256	1550	0.025-0.256	27.2345×10^5	11.83	396.16
	4) 0-290	1459	0.029-0.290	42.80852×10^5	12.40	387.88

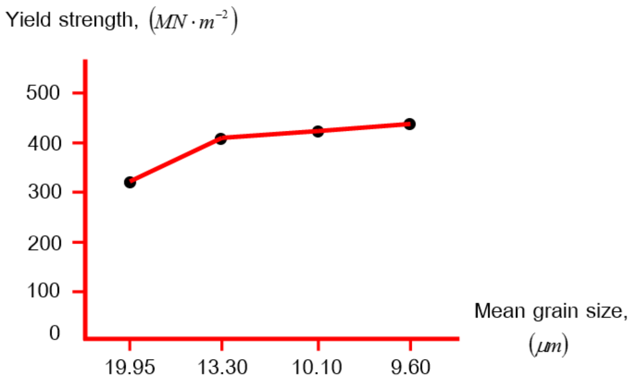

Figure 4. Effect of mean polygonal acicular ferrite grain size (mm) on yield strength $(MN \cdot m^{-2})$ from the best microalloyed steel as ME Wrench 1 in Table 3.

The following Hall-Petch relationships can be expressed for the microalloyed steel (Kazeminezhad, 2004) and (Gladman, 1997)

$$\sigma_y = 40 + 1.225 \quad d^{-0.5} \tag{6}$$

where σ_y and *d* are yield strength (MN m^{-2}) and mean ferrite grain size (mm), respectively. In Table 4 the mean ferrite grain size as determined by Spektor's method is compared to values of G, d, and l estimated using International Standard (ISO 643) (International Standard, 2003) as a function of various parameters. The values of mean ferrite grain size ranged from 09.60 to 20.65 µm, and the agreement between the two methods is quite good. The evaluation mean polygonal-acicular ferrite grain size of ME Wrench 1 is 19.95, 13.30, 10.10, and 09.60 µm, ME Wrench 2 is 21.0, 13.50, 10.63 and 10.11 µm, ME Wrench 3 is 20.30, 13.59, 11.63 and 12.14 µm, and ME Wrench 4 is 20.65, 14.10, 11.83 and 12.40 µm that showed a secondary structure response. Vanadium is known to promote precipitation strengthening and makes smaller mean grain size than in steels without it. The mean grain size of ferrite in steel ME Wrench 1, (row 1, Table 3 as 19.95 µm), is the largest because it has 0.121 V, 0.0039 Ti, and 0.00045 Nb of micro-alloyed elements. The double micro-alloyed steel ME Wrench 1, (row 4, Table 3 as 09.60 µm), exhibited the smallest ferrite grain size. Figure 5 shows yield strength ($MN.m^{-2}$) bar for present ME Wrench 1 from Table 2 and 3 at the given Vanadium or Niobium content (wt%) additions.

As shown in Figure 6, hardness distributions of each wrench are different, because these distributions are created a U shape area is the measured position of these four wrenches (Table 2 and Figure 1). The hardness examination of the investigated drop forging microalloyed HSLA steels shows that ME Wrench 1 is about 131, 165, 179, and 197 HB, ME Wrench 2 is about 110, 159, 175, and 185 HB, ME Wrench 3 is about 120, 155, 162, and 175 HB and ME Wrench 4 is about 118, 162, 170, and 175 HB, these steels present significant differences in hardness as shown in Figure 6. These hardness results can be compared to results of hardness measurements by Skubisz *et al.* (2008), see Figure 8.

Table 4. Evaluation mean grain size of Spektor's method with International Standard (ISO643) [International Standard (ISO643), 2003].

1 Steel	2 Evaluated mean grain size, µm, d	3 Estimated grain size (Index), G	4 Mean diameter of grain, µm, đ	5 Mean intersected segment, µm, I
ME Wrench 1	1) 19.95	8.5	18.7	14.2
	2) 13.30	9	12.1	11
	3) 10.10	9.5	9.8	9.5
	4) 09.60	10	8.5	8.2
ME Wrench 2	1) 21.0	8	20.5	18.8
	2) 13.50	9	12.5	11.2
	3) 10.63	9.5	10.2	9.8
	4) 10.11	9.5	9.8	9.5
ME Wrench 3	1) 20.30	8.5	18.45	17.35
	2) 13.59	9	12.3	11.3
	3) 11.63	9.5	10.25	9.25
	4) 12.14	9.5	11.15	10.8
ME Wrench 4	1) 20.65	8.5	18.5	17.3
	2) 14.10	9	13.9	12.5
	3) 11.83	9.5	9.9	8.5
	4) 12.40	9.5	10.2	9.8

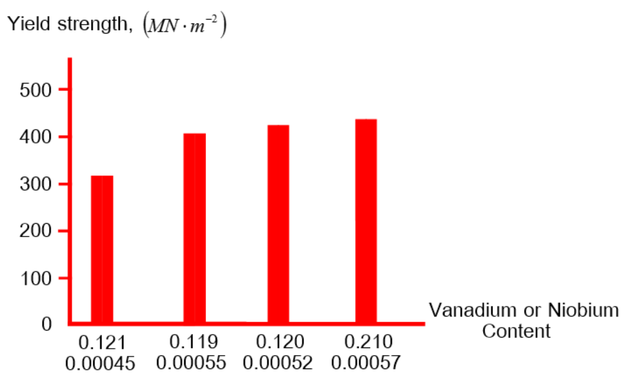

Figure 5. Yield strength $(MN \cdot m^{-2})$ bar for present ME Wrench 1 from Table 2 and 3 at given Vanadium or Niobium content (wt%) additions.

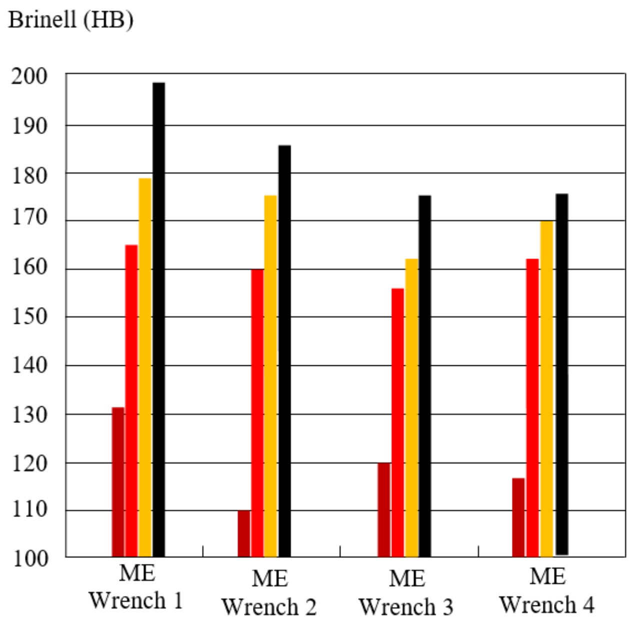

Figure 6. Hardness distributions (HB) of the four drop forging microalloyed HSLA steels.

4. Conclusions

1. The Hall-Petch relationship can be utilized to examine the dependence of yield strength on mean ferrite grain size of the microalloyed HSLA steels. One method of controlling the properties of steel is by controlling the grain size. By reducing the grain size, increase the number of grains and the amount of grain boundary. Any dislocation moves only a short distance before encountering a grain boundary, and the strength of the steels are increased.

2. Vanadium and Niobium as microalloying elements increase the yield strength and decrease the mean ferrite grain size significantly also in as cast state.

3. The investigated four drop forging microalloyed HSLA steels have a ferritic-pearlitic structure. However, their microstructures are quite different. ME Wrench 1 with high vanadium content 0.121, 0.119, 0.120, and 0.210% and niobium 0.00045, 0.00055, 0.00052, and 0.00057% has a much higher volume fraction of proeutectoid ferrite which forms a discontinuous network along the prior austenite grain boundaries.

4. Microhardness of proeutectoid ferrite measured for ME Wrench 1 is about 131, 165, 179, 197 HV0.2 higher than for other three group steels.

5. Based on the results of the investigations carried out, it has been found that increase of vanadium and niobium contents have a significant influence on the microstructures. Better mechanical properties are expected, which will confirm the structural benefits characteristic for ME wrench 1 steel.

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