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Estimation of the mean size of the cementite particles by hardness measurement in plain carbon steel

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The relationship between the Vicker's hardness VHN and the mean size of the coarsening cementite particles, $\bar{d}$ in plain carbon steel during tempering at high temperature has been deduced and corroborated by experimental data analysis. It is found that VHN varies linearly with $1/\bar{d}$. Hardened plain carbon steel with 0.68, 0.56 and 0.34% carbon, has been tempered at 973 to 861 K as a function of time for the investigation. A master plot of VHN against $\bar{d}(1 - V_v)/V_v$ has been drawn which provides the mean particle diameter of the cementite particles as a function of hardness, irrespective of time and temperature of tempering and carbon content of the steel.

1. Introduction

Mean particle size has been measured [1–20] as a function of tempering parameters by various researchers to study the kinetics of Ostwald ripening by the direct measurement using either electron microscope or optical microscope. The direct measurement, however, is a very tedious process. Various problems are faced in sample preparation [21] and measurement of particles [2, 3, 5, 7, 19, 20]. Measurements on a large number of particles are required and various errors [22] encountered.

Measurement of mean particle size, therefore, needs a simple and convenient method and should be versatile enough so that it can be used to evaluate deterioration or suitability of a component during service under industrial condition. The authors have not found any reference to any work of this nature in the literature. The present work is an attempt to meet such a need for plain carbon steel.

In the present work the relationship between $\bar{d}$ and VHN has been deduced and this relationship in the form of a master plot has been substantiated by experimental data analysis and used for the estimation of $\bar{d}$ from hardness in tempered plain carbon steel.

2. Theoretical analysis

A straight line parallel to the X axis of a unit cube containing N_v number of spherical particles with an average diameter $\bar{d}$ is considered. The probability that the line will intersect a specific sphere equals the projected area of the mean sphere on the X plane and is given by $\pi\bar{d}^2/4$. Hence the number of interceptions of the particles per unit length of test line $\bar{N}_L$ is given by

$$\bar{N}_L = \frac{\pi\bar{d}^2}{4} N_v \quad (1)$$

The total number of intersections $\bar{N}_A$ with all particles in the structure formed with the unit area of a randomly oriented test plane is given [22] by

$$\bar{N}_A = N_v \bar{D}_v \quad (2)$$

where $\bar{D}_v$ is the average distance between tangent planes of all the particles. For a polydispersed system of spheres Equation 2 reduces [23] to

$$\bar{N}_A = \bar{d} N_v \quad (3)$$

where $\bar{d}$ is the mean particle diameter. From Equations 1 and 3

$$\bar{d} = \frac{4}{\pi}\frac{\bar{N}_L}{\bar{N}_A} \quad (4)$$

From Equation 3

$$N_v = \frac{\bar{N}_A}{\bar{d}} \quad (5)$$

Lifshitz and Slyozov [24] and Wagner [25] in their pioneering work have developed the theory of diffusion controlled particle coarsening. Martin and Doherty [1] following Greenwood have developed an equivalent equation for Ostwald ripening. If a narrow Gaussian particle size distribution is assumed, the rate equation for particle coarsening [1] where the distribution has a sharp cut off at $r = 1.5\bar{r}$ has been achieved ($\bar{r} = \bar{d}/2$). For a narrow particle size distribution, the volume V_v of the coarsening particles in a unit cube containing N_v number of spherical particles with an average diameter $\bar{d}$ is given by

$$V_v = \tfrac{4}{3}\pi\left(\frac{\bar{d}}{2}\right)^3 N_v$$

$$= \frac{\pi\bar{d}^3}{6} N_v \quad (6)$$

From Equations 4, 5 and 6

$$V_v = 0.849\frac{\bar{N}_L^2}{\bar{N}_A} \quad (6a)$$

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Equation 6 has been substantiated by comparing results obtained by Equation 6a with those obtained by another independent method namely the point counting method [22]. This method has been taken for granted as the best method of V_v measurement. It uses the relationship

$$V_v = P_p = \frac{P_\alpha}{p_T} \quad (6b)$$

where P_α is the number of test points falling on the phase α and P_T is the total number of test points.

From Equations 1 and 6

$$\bar{d} = \frac{3}{2}\frac{V_v}{\bar{N}_L} \quad (7)$$

The mean free path is given by

$$\bar{\lambda} = \frac{1 - V_v}{\bar{N}_L} \quad (8)$$

or

$$\bar{N}_L = \frac{1 - V_v}{\bar{\lambda}}$$

From Equations 7 and 8

$$\bar{d} = \tfrac{3}{2}\bar{\lambda}\frac{V_v}{1 - V_v} \quad (9)$$

Tabor's method [26, 27] to obtain the plastic region of the true stress–true strain curve from the Meyer [28] hardness measurement showed agreement between the flow curve and Meyer hardness against d/D curve for mild steel and copper where d/D is the ratio of diameter of indentation to diameter of ball. In agreement with Tabor's results, the 0.2% offset yield strength is given [29] as

$$\sigma_0 = \frac{\text{VHN}}{3}(0.1)^{n'-2} \quad (10)$$

where σ_0 is the 0.2% off-set yield strength in kg mm^{-2}, VHN the Vickers hardness number and n' the exponent in Meyer's [28] law.

The increase in shear stress ($\Delta\tau$) of a metal having precipitates with mean planar separation $\bar{\lambda}$, due to bowing of dislocations having line tension T_1 is given by the Orwan [30] equation

$$\Delta\tau = \frac{2T_1}{b\lambda} \quad (11)$$

where b is the Burgers vector. Equations 10 and 11 give

$$\text{Strength} \propto \text{VHN} \quad (12)$$

$$\text{Increase in strength} \propto \frac{1}{\bar{\lambda}} \quad (13)$$

$$\text{Hence increase in VHN} = K_1\frac{1}{\bar{\lambda}} \quad (14)$$

From Equations 9 and 14

$$\text{VHN} = H_0 + K_2\frac{V_v}{1 - V_v}\cdot\frac{1}{\bar{d}} \quad (15)$$

TABLE I Composition of the steels

Steel	C (wt %)	Si (wt %)	Mn (wt %)	S (wt %)	P (wt %)
A	0.68	0.23	0.45	0.024	0.015
B	0.56	0.30	0.62	0.024	0.034
C	0.34	0.16	0.50	0.025	0.017

where H_0 is the VHN of the material when $\bar{\lambda} = \infty$; i.e. no precipitate, and K_1 and K_2 are constants.

3. Experimental procedures

Three hypoeutectoid plain carbon steels were used for this work. The chemical analysis of the steels is given in the Table I. Samples prepared from the steels A, B and C were hardened by water quenching. The hardened samples were isothermally tempered in a lead bath at temperatures 973, 953, 930 and 861 K as a function of time for 1.5 to 209 h and the hardness (VHN) of the samples was measured with a 30 kg load. The bath temperature was controlled to ± 1 K. The tempered samples were prepared for microstructural observation. The microstructure consists of nearly spherical cementite particles in the matrix of ferrite (Fig. 1).

The chemical analysis of the steels A, B and C given in Table I shows that they do not contain any strong carbide forming elements and the effect of other alloying elements, which are present to a small amount in these steels, on the composition of carbides, is expected to be very small. Thus the carbides formed in these steels are taken to be Fe_3C.

Micrographs have been taken at random for each sample at a convenient magnification for statistical measurement. The mean carbide particle diameter $\bar{d}$, volume fraction V_v of the coarsening particles and mean free path $\bar{\lambda}$ have been estimated by lineal and areal density analysis using the relationships given by Equations 4, 6a and 8 respectively. A rectangular grid has been used for the estimation of the number of interceptions of the particles per unit length of the test line $\bar{N}_L$ and the number of particles per unit test area $\bar{N}_A$. The parameters $\bar{N}_L$ and $\bar{N}_A$ have been subsequently used to estimate the stereological parameters.

4. Results and discussion

The relative values of $\bar{d}$, V_v, $\bar{\lambda}$ and VHN are given in

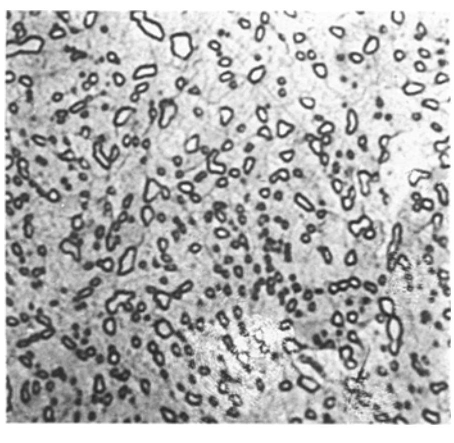

Figure 1 Micrograph of 0.68% steel tempered at 953 K for 24 h.

TABLE II Relative values of V_v, $\bar{d}$, $\bar{\lambda}$ and VHN

C (wt % of steel)	$V_v \times 100$	$\bar{d}$ (μm)	$\bar{\lambda}$ (μm)	VHN 30 kg load
0.68	11.6	0.58	2.96	216
	11.6	0.63	3.18	204
	11.8	0.71	3.53	189
	11.7	0.77	3.88	184
	11.8	0.57	2.85	203
	11.8	0.63	3.15	197
	11.3	0.69	3.62	189
	11.8	0.72	3.59	186
	11.8	0.76	3.81	183
	11.8	0.88	4.40	172
	12.0	0.52	2.54	248
	12.0	0.55	2.68	234
	11.9	0.59	2.89	226
	12.9	0.63	2.84	212
	12.8	0.65	2.96	207
0.56	10.8	0.54	2.97	217
	10.3	0.62	3.60	201
	10.5	0.69	3.92	195
	10.0	0.87	5.18	185
	10.1	0.57	3.38	211
	9.8	0.68	4.16	194
	10.0	0.78	4.66	185
	10.0	0.85	5.12	184
	10.2	0.47	2.75	232
	10.2	0.51	3.00	219
	10.1	0.56	3.34	211
	9.9	0.58	3.52	206
0.34	5.8	0.46	5.01	176
	6.0	0.49	5.11	173
	5.9	0.52	5.72	161
	6.1	0.58	6.04	163
	5.7	0.44	4.73	176
	6.0	0.46	4.85	175
	6.0	0.50	5.21	167
	6.4	0.53	5.18	163
	6.0	0.59	6.18	162
	5.5	0.64	7.38	154
	5.9	0.39	4.08	200
	6.0	0.40	4.12	197
	5.5	0.44	5.04	190
	6.0	0.45	4.74	185
Samples tempered in different steps of time and temperature				
0.68	11.6	0.73	3.72	182
0.56	10.4	0.72	4.10	185
0.34	5.9	0.56	5.90	152

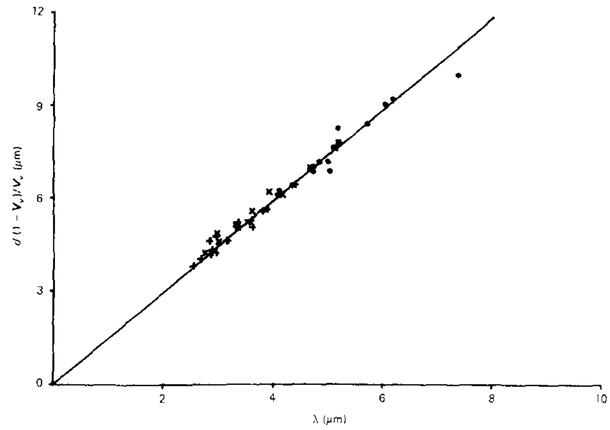

Figure 2 Plot of $\bar{d}(1 - V_v)/V_v$ $\bar{\lambda}$ for different steels tempered at different temperatures. (+, 0.68% C steel; x, 0.56% C steel; *, 0.34% C steel).

Table II. To check the consistency of the volume fraction measurement by Equation 6a, the volume fraction of carbides in some of the samples has also been measured by the point counting method using Equation 6b. The values of V_v measured by these two sets of independent linear and area density method and point counting method have shown a good agreement as given in Table III. This table also shows the theoretical values of V_v which have been calculated from the carbon content of the steels and iron–carbon equilibrium diagram. The values of $\bar{d}$ and N_v estimated by Equation 5 are also reported in Table III.

Equation 9 shows $\bar{d}$ as a linear function of $\bar{\lambda}[V_v/(1 - V_v)]$ which has been substantiated by linear regression analysis of the experimental data. A correlation coefficient of almost unity (0.999) suggests a good fit of the experimental data to Equation 9. This is once again depicted by Fig. 2. The plot shows an increase of $\bar{\lambda}$ with an increase of $\bar{d}$ during the tempering of steels.

Equation 14 expresses VHN as a linear function of $1/\bar{\lambda}$. Linear regression analysis of experimental data of VHN against $1/\bar{\lambda}$ has produced a correlation coefficient nearly equal to unity (0.917) and the plot of VHN against $1/\bar{\lambda}$ given in Fig. 3 shows a good fit of the data to Equation 14. This may be taken as the experimental verification of the relationship.

TABLE III Comparison of volume fraction as determined from carbon content, linear–area density analysis and point count.

Sample number	V_v from C %		$V_v = 0.849\ N_L^2/\bar{N}_A$		$V_v = P_\alpha/P_T$		$\bar{d}$ (μm)	$N_v \times 10^{-8}$ (numbers mm^{-3})
	C (wt %)	V_v (%)	V_v (%)	ΔV_v	V_v (%)	ΔV_v		
56A′1	0.56	8.5	10.8	0.02	9.9	0.03	0.54	13.10
56A′2	0.56	8.5	10.3	0.02	9.9	0.03	0.62	8.27
56A′3	0.56	8.5	10.5	0.02	9.9	0.03	0.69	6.04
56A′4	0.56	8.5	10.0	0.02	10.8	0.03	0.87	2.94
56B′4	0.56	8.5	10.0	0.02	9.9	0.03	0.85	3.07
68B′4	0.68	10.4	11.7	0.02	11.7	0.03	0.77	4.85

56A′1 – 0.56 wt % C steel tempered at 973 K for 1.5 h
56A′2 – 0.56 wt % C steel tempered at 973 K for 4 h
56A′3 – 0.56 wt % C steel tempered at 973 K for 8 h
56A′4 – 0.56 wt % C steel tempered at 973 K for 24.5 h
56B′4 – 0.56 wt % C steel tempered at 953 K for 49.25 h
68B′4 – 0.68 wt % C steel tempered at 953 K for 24 h

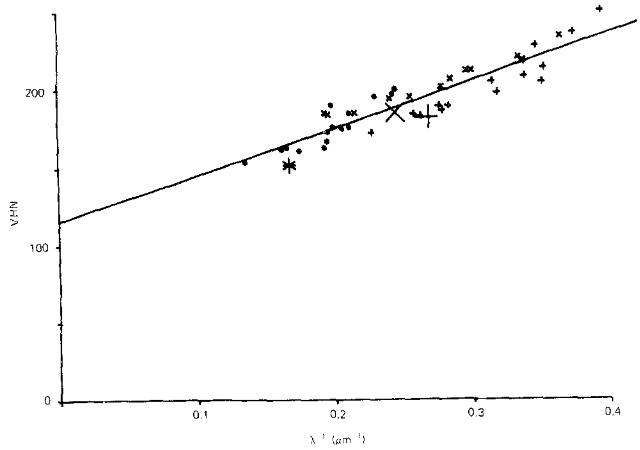

Figure 3 VHN as a linear function of $1/\bar{\lambda}$. Bigger symbols indicate data for step tempered samples. (+, 0.68 % C steel; x, 0.56% C steel; $*$, 0.34 % C steel).

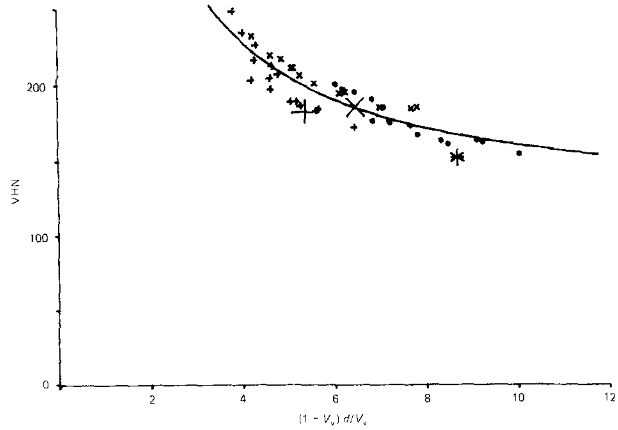

Figure 4 VHN as a function of $(1 - V_v)\bar{d}/V_v$ for different steels tempered at different temperatures. Bigger symbols indicate data for step tempered samples. (+, 0.68% C steel; x, 0.56 % C steel; $*$, 0.34 % C steel).

Fig. 3 shows that VHN decreases with increase of $\bar{\lambda}$ and it may reach its lowest value of 116 VHN at $\bar{\lambda} = \infty$ i.e. hardness of ferrite at its equilibrium concentration of carbon.

The experimental data fit well to both Equations 9 and 14 and these two equations yield Equation 15 which relates VHN with $\bar{d}$. The validity of Equation 15 has been further checked by linear regression analysis of VHN against $[V_v/(1 - V_v)](1/d)$ data (experimental). The results of the regression analysis are reported in Table IV. The near unity correlation coefficient (CC = 0.915) indicates good fit of the experimental data to Equation 15.

Fig. 4 gives VHN as a function of $\bar{d}$ and volume fraction and it reveals how well the experimental $\bar{d}$ and VHN match the calculated value, whereas the mathematical fit of the same quantities has been depicted by the linear regression analysis through Equation 15. It may be noted that though the error in inverse relationship between VHN and $\bar{d}$ has been minimized, the curve in Fig. 4 shows the error in $\bar{d}$ and VHN is still quite low. The curve is a rectangular first degree hyperbola and it reveals the deterioration of material during Ostwald ripening through the decrease of hardness with the progress of particle coarsening.

All three plots in Figs 2, 3 and 4 accommodate all the data obtained as a function of tempering time for all the steels to produce master plots which are independent of temperature. One sample from each of the steels A, B and C has been tempered in three steps with three different combinations of time and temperature. The steps for samples obtained from steels A (0.68 % C) and B (0.56 % C) are 12.5 h at 955 K, 26 h at 929 K and 42 h at 877 K and for the sample obtained from steel C (0.34 % C) are 6 h at 956 K, 21 h at 932 K and 25 h at 875 K. The relative values of the experimental parameters are shown in Table II. Figs 3 and 4 show that the data of the step-tempered samples agree well with the plots. The plots of VHN against $1/\bar{\lambda}$ and VHN against $\bar{d}(1 - V_v)/V_v$ are dependent only on the final mean values of the data. The most interesting feature of the plot of VHN against $\bar{d}(1 - V_v)/V_v$ is that with a known volume fraction V_v (which can be expressed as a function of composition for tempered steel) the mean particle diameter $\bar{d}$ can be found from the plot by a relatively simple hardness (VHN) measurement.

TABLE IV CC, slope and intercept for the linear regression analysis of VHN against $[V_v/(1 - V_v)](1/\bar{d})$

CC	Slope VHN (μm^{-1})	Intercept VHN
0.915	4.65×10^2	1.14×10^{-2}

Plain carbon steel is widely used for the manufacture of boiler and pressure vessels. Components used at elevated temperatures quite often fail due to deterioration of the material by the growth of precipitate size. The simple method of estimating $\bar{d}$ from VHN may prove to be of great help to maintenance engineers to guard against component failure at elevated temperature.

The steels used in this investigation are commercial grade steels and their austenitizing temperatures selected are the ones which are commonly employed on the shop floor. The prior austenitic grain size and ferrite (tempered martensite) grain size are likely to affect the hardness. However in the present work these effects have not been accounted for. Yet as all the data give unified relationships, it may be concluded that the effect of grain size is insignificant compared to contributions of size and volume fraction of carbide particles.

5. Conclusions

(1) Estimation of particle size by hardness (VHN) measurement is a simple and convenient method which is relatively free from personal error.

(2) The relationship between VHN and $\bar{d}$ is independent of history of tempering of the hardened plain carbon steel.

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