

# Effect of matrix structure and mechanical deformation on carbide coarsening

By W. E. STUMPF,\* B.Sc. Ing. Met., Ph.D. (Member) and C. M. SELLARS,† B.Met., Ph.D. (Visitor)

## SYNOPSIS

The effects of concurrent recovery, prior creep deformation and concurrent high temperature fatigue on the coarsening of some carbides in steels have been studied. It was found that concurrent recovery enhances the coarsening rate by orders of magnitude and prior creep deformation causes an enhancement of about two times. Finally, concurrent fatigue causes an initial greatly enhanced coarsening rate but after prolonged cycling dissolution of the carbides occurs.

## INTRODUCTION

Most of the present high temperature alloys are of the precipitation-hardened type. In these alloys the coarsening of the second phase particles at service temperatures, usually accompanied by a decrease in high-temperature strength, can be significant. Coarsening of the larger carbide particles occurs by the diffusion of carbon, which becomes available due to the dissolution of the thermodynamically unstable smaller particles. The kinetics of the coarsening process can be predicted by the formula

$$d^3 - d_0^3 = \frac{64 \gamma D Co V_m^2 t}{81 RT}$$

This formula is based on theoretical considerations discussed by Lifshitz and Slyozov<sup>1</sup> and by Wagner<sup>2</sup> for a diffusion-controlled mechanism of growth.

The symbols used in the above equation have the following meanings:

- $t$  = time.
- $T$  = temperature °K
- $d$  = mean particle size at time  $t$ .
- $d_0$  = original particle size.
- $\gamma$  = particle-matrix interfacial energy.
- $Co$  = solubility limit of solute at Temperature  $T$ .
- $V_m$  = molar volume of precipitate.
- $D$  = diffusion coefficient and
- $R$  = the gas constant.

Prior or concurrent deformation can alter the kinetics of coarsening significantly by enhancing the inter-particle diffusivity or by increasing the solubility limit, thus causing an accelerated drop in strength under such service conditions. This paper summarizes some results<sup>3-6</sup> of the effects of mechanical deformation on the coarsening characteristics of carbides in a plain carbon steel and some alloy steels.

## EXPERIMENTAL TECHNIQUES

The chemical analysis of the steels used in this investigation are shown in Table I. Column 3 of this table shows the equilibrium carbide for each composition.

These steels were quenched to martensite and then tempered at 700°C for various times. Coarsening of the carbides under static conditions was followed in these specimens. In a few cases the steels were only tempered for a short time after quenching and then cold swaged and retempered at 700°C for longer times. This produced a fully recrystallized structure in contrast to a

TABLE I  
ANALYSES OF STEELS

Carbon %	Chromium %	Carbide
0.20	0	Fe <sub>3</sub> C
0.21	0.87	M <sub>3</sub> C
0.21	1.2	M <sub>3</sub> C + M <sub>7</sub> C <sub>3</sub>
0.21	2.4	M <sub>7</sub> C <sub>3</sub>
0.20	4.2	M <sub>7</sub> C <sub>3</sub>
0.20	6.1	M <sub>7</sub> C <sub>3</sub>
0.21	11.7	M <sub>23</sub> C <sub>6</sub>

fully recovered structure after tempering of the simply quenched steels (Hereafter referred to as recrystallized and as-tempered steels respectively.)

The effect of concurrent recovery on the coarsening behaviour of cementite could be followed after short tempering times in the as-tempered plain carbon steel<sup>3</sup>. This was possible as recovery of the high dislocation contents, introduced by the martensitic transformation, occurred relatively slowly upon subsequent tempering. This allowed sufficient time for experimental observations before completion of the recovery. Secondly, the effect of a prior high temperature creep deformation on the coarsening of cementite was studied by creep deforming a recrystallized as well as an as-tempered structure prior to subsequent tempering<sup>4, 5</sup>. Finally, the effect of a concurrent high temperature fatigue deformation on the coarsening of carbides was studied on the plain carbon steel as well as on most of the alloy steels<sup>6</sup>. These deformations were carried out at 700°C at different stresses and frequencies and the coarsening behaviour was followed by interrupting the fatigue test after various numbers of cycles.

Particle size measurements were made from shadowed extraction replicas using the procedure described and assessed elsewhere<sup>3, 7</sup>. Some volume fraction determinations of carbides in the fatigued steels were also carried out using a double replica technique<sup>8</sup>.

## RESULTS AND DISCUSSION

### Concurrent recovery

Fig. 1 shows the coarsening behaviour of cementite in the as-tempered plain carbon steel at 700°C. In this

\*Scientist, Physical Metallurgy Division, Atomic Energy Board.  
†Senior Lecturer, Department of Physical Metallurgy, University of Sheffield, United Kingdom on a year's leave of absence at The Broken Hill Prop. Co. Ltd., Melbourne Laboratories, Australia.

figure  $\bar{X}_{G2}$  is the geometric mean volume particle diameter. The coarsening is very rapid until about 5 hours at 700°C where a marked reduction in coarsening rate occurs. Even so, this slower coarsening rate is still noticeably more rapid than in the steel recrystallized after cold work, also shown on the same figure.

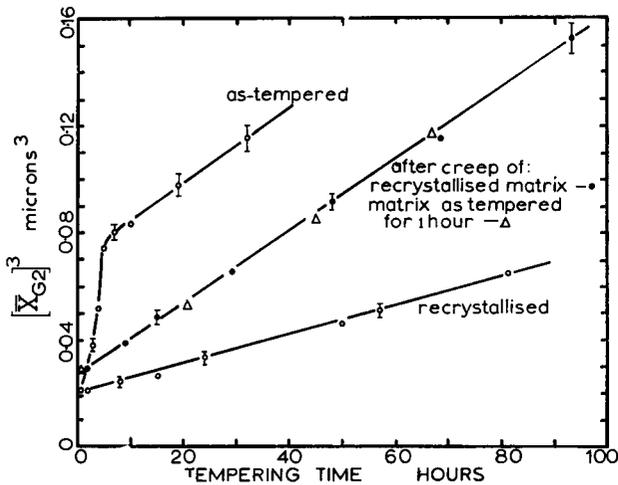


Fig. 1—Relation between the cube of geometric mean particle diameter and tempering time at 700°C for an 0.21% plain carbon steel with different matrix structures.

During the early stages of tempering recovery of the high dislocation content (introduced by the martensitic transformation) takes place and this causes a rapid rate of coarsening. After about 5 hours at 700°C the recovery is complete and a well developed substructure, linking most of the particles, is found, as shown in Fig. 2(a). In Fig. 2(b) the dislocation arrangement is shown in the steel recrystallized after cold work. In this case the low dislocation density probably causes negligible enhancement of coarsening and consequently the coarsening rate in this matrix structure can be taken as representative of the true unenhanced coarsening rate. The substructure in Fig. 2(a) was found to be quite stable upon further tempering at 700°C except for a small degree of subgrain growth at longer times. During this stage of tempering the enhancement in coarsening rate is less than when moving dislocations are present but is still enhanced by the presence of subgrains linking adjacent particles.



Fig. 2a

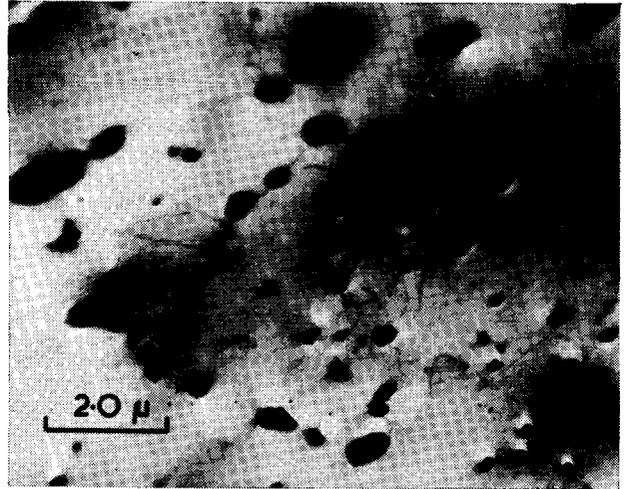


Fig. 2b

Fig. 2—Thin foil micrographs of an 0.21% plain carbon steel (a) tempered 10 hours at 700°C and (b) tempered one hour, then cold swaged 27% and re-tempered four hours at 700°C to recrystallize the matrix.

This preliminary work indicated that significant enhancements in coarsening rate can be expected if stable microstructural changes are introduced into the matrix and that the enhancement can become very large if the microstructural changes are unstable or are introduced dynamically. Little can, however, be said about the mechanism of enhancement in each case. This can only be done by introducing quantitative microstructural changes into the matrix and observing the effect these have on the mechanism of coarsening itself.

#### Prior creep deformation

Many metals form a subgrain network during steady state creep at high temperatures. The size of the subgrains ( $\lambda$ ) is a unique function of the applied creep stress ( $\sigma$ ) and is given by

$$\lambda = K/\sigma \dots\dots\dots(2)$$

where  $K$  is a constant which depends, to some extent, on the stacking fault energy of the metal.

This technique was used to introduce a stable substructure into the plain carbon steel prior to further static tempering, during which the slightly accelerated coarsening could be followed. A relatively high creep stress of 10 500 lb/in<sup>2</sup> at 700°C was used to introduce a fine subgrain size, as predicted by equation 2. Practically all the particles are thus linked by subgrain boundaries, as shown by the thin foil electron micrograph in Fig. 3. Coarsening of cementite in such a substructure is included in Fig. 1 for prior creep deformation of the as-tempered as well as the recrystallized structures.

It is evident that, once again, the coarsening rate is enhanced to the same degree as was found in the as-tempered structure after about 5 hours tempering time at 700°C. Furthermore, it is interesting to note that a prior creep deformation of an as-tempered structure eliminates the slow recovery upon subsequent tempering that was found in the as-tempered steel at times less than 5 hours. The substructure, formed by creep, was also found to be relatively stable upon subsequent tempering as shown in Fig. 4. The subgrain size was measured by a back-reflection X-ray microbeam technique and the interparticle spacing calculated from the particle size distributions<sup>9</sup>.



Fig. 3—Thin foil micrograph of an 0.21% plain carbon steel recrystallized as in Fig. 2(b) and then creep deformed 11% at 10 500 lb/in.<sup>2</sup> and 700°C.

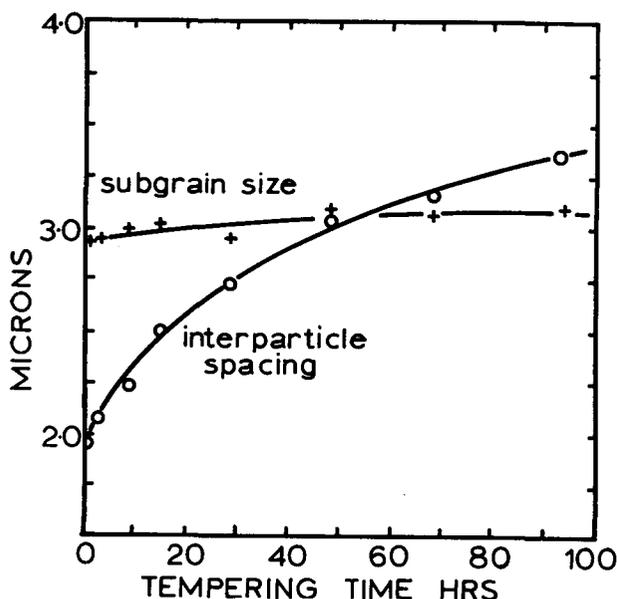


Fig. 4—Change in subgrain size and interparticle spacing in recrystallized and creep deformed 0.21% plain carbon steel on subsequent tempering at 700°C.

To establish the mechanism of enhancement in a stable substructure, the activation energy for coarsening was determined for unenhanced coarsening in a recrystallized matrix and was compared with that for enhanced coarsening in a recrystallized and then creep deformed matrix. Additional coarsening rates were determined at 600 and 650°C and from the appropriate Arrhenius plots, the activation energies for diffusion were found for the two cases. From equation 1 it can be seen that for a unique activation energy for diffusion to be determined, the coarsening rate ( $K$ ) has to be compensated by the temperature dependence of  $C_0$  and  $T$  itself. Thus in the case of unenhanced coarsening, a plot of  $\log (TK/C_0)$  vs.  $1/T$  yielded an activation energy for diffusion of 60 Kcal/mole. This indicates that normal coarsening of cementite is controlled by the diffusion of vacancies in ferrite and not by the diffusion of carbon in which case the activation energy would

have been about 20 Kcal/mole. In the case of enhanced coarsening  $K$  could not be compensated for by the temperature dependence of  $C_0$  but  $C_b$  had to be used, where  $C_b$  is the much larger solubility limit of carbon on a subgrain boundary. This yielded an activation energy of 58 Kcal/mole which, once again, agrees well with the activation energy for lattice self diffusion in ferrite and does not agree with the activation energy for grain boundary self diffusion in ferrite of 40 Kcal/mole<sup>10</sup>.

It appears, therefore, that enhanced coarsening of cementite on a stable substructure between 600 and 700°C, is not caused by enhanced diffusion along the sub-boundaries linking the particles, but is apparently due to the greater solubility of carbon on a subgrain boundary. Such an enhancement must necessarily be a critical function of the binding energy between solute atoms and dislocations. Much smaller effects would thus be expected for particles of inter metallic compound when only substitutional elements are involved and the binding energies are much smaller. This may account for the lack of effect of concurrent creep on the coarsening rate of  $\gamma'$  in nickel base alloys<sup>11</sup> compared with large enhancements found for carbides in steel during concurrent creep<sup>12</sup> or tensile deformation<sup>13</sup>.

#### Concurrent high temperature fatigue

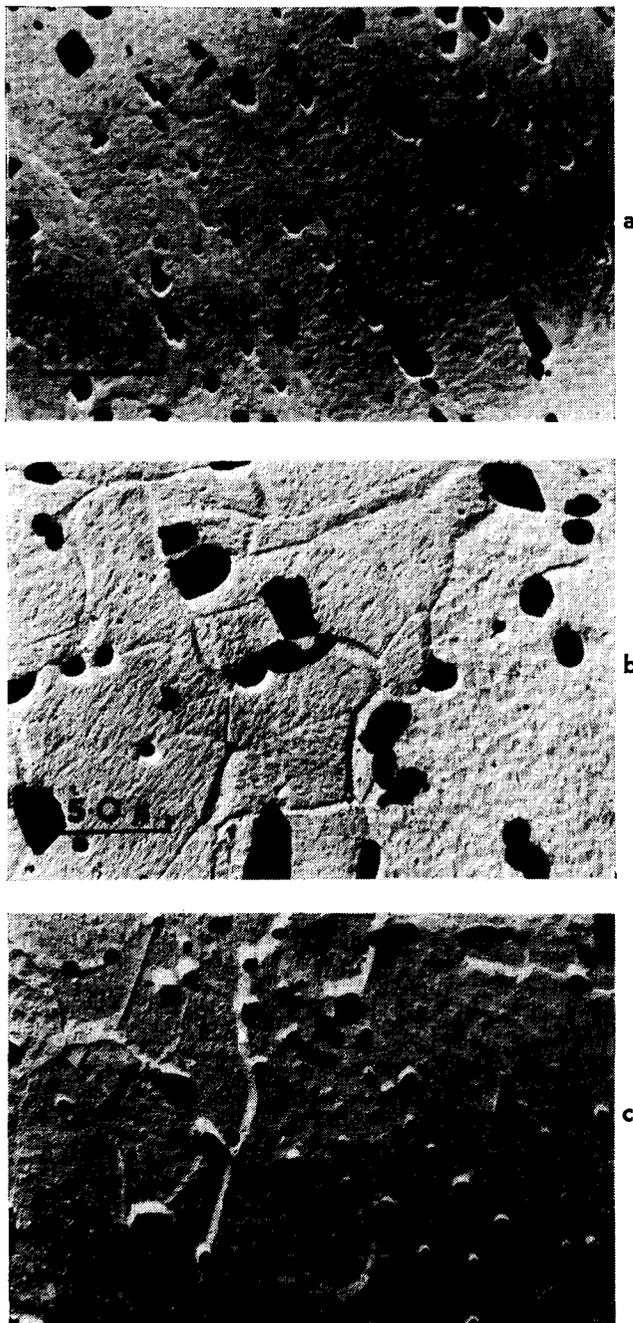
High temperature fatigue can produce a large number of structural changes in precipitation hardened alloys. These include: overaging or enhanced coarsening of the precipitates, the promotion of incoherency between particles and the matrix, the dissolution of precipitates, the nucleation of new precipitates and an increase in solid solubility limit of solute atoms. Most of these observations, however, were qualitative only and little could be said about the operative mechanisms involved. In this investigation some quantitative particle size and volume fraction measurements of the carbides in these steels, undergoing concurrent high temperature fatigue, were carried out and analyzed critically.

Shadowed extraction replicas during fatigue at 700°C, are shown in Fig. 5 (a) to (c). During the first 15 000 cycles a rapid increase in particle size occurs, but upon further cycling many smaller particles re-appear and the mean particle size decreases again. Furthermore, in these structures practically all particles are linked by subgrain boundaries during the early stages of fatigue but upon further cycling many new particles appear within the subgrains, indicating that these could possibly be freshly nucleated carbides.

Fig. 6 shows the dislocation structure in a recrystallized plain carbon steel after fatigue at 700°C. The initially low dislocation density (Fig. 2 (b) ) increases during fatigue to a very uniform dislocation structure with relatively few dislocation tangles. A number of dislocation loops, most of which are elongated, can also be seen but no intense dislocation-particle interaction and no dislocation cell or band structure was observed.

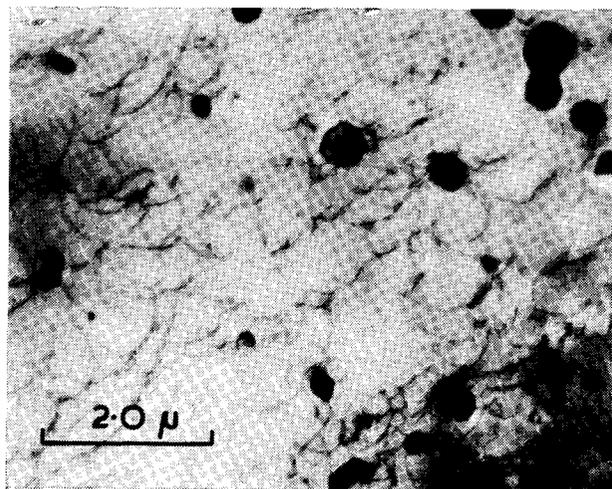
The change in mean particle size during fatigue of the recrystallized 0 and 0.87 per cent Cr steel is shown in Fig. 7 (a) while in Fig. 7 (b) the corresponding strain per cycle at the constant stress of 10 500 lb/in.<sup>2</sup> is shown. Both during the initial increase and during the subsequent decrease in particle size, a bodily shift of the distribution curves occurred without the shape of the curve being largely affected. This indicates that the distribution as a whole is undergoing change and not only, for instance, smaller particles, as would be the

case if repeated cutting of the small particles by moving dislocations caused them to dissolve. During the initial period of fatigue, when work hardening of the specimens took place as shown in Fig. 7 (b), the coarsening rates were enhanced by factors typically between 1 000 and 10 000 depending on the matrix structure, type of carbide present and also, to some extent, on the fatigue frequency and stress used.



**Fig. 5—Carbide structures in 1.2% Cr steel after 1 000 hours tempering at 700°C and then fatigue cycling at 10 500 lb/in<sup>2</sup>, 700°C and 45 cycle/sec for (a) 0 cycles, (b) 15 000 cycles and (c) 50 000 cycles.**

The change in volume fraction of carbide during fatigue was determined for the recrystallized plain carbon steel and is shown in Fig. 7 (c). During the first 10 000 cycles, when rapid coarsening of the cementite particles occurs, the volume fraction remains constant and close to the equilibrium value. Upon further cycling, however, the volume fraction decreases significantly, caused by the dissolution of particles. Analyses showed that this decrease in volume fraction is not due to decarburization of the specimen. The only other alternative is an increase in carbon solubility limit of the matrix. From the measured volume fractions in the plain carbon steel it was calculated that this would have to increase from about 0.02 per cent carbon during the first 10 000 cycles to about 0.05 per cent after 75 000 cycles of fatigue.



**Fig. 6—Thin foil micrograph of recrystallized plain carbon steel after fatigue at 10 500 lb/in<sup>2</sup>, 700°C and 45 cycle/sec for 75 000 cycles.**

The behaviour of individually sized particles in a distribution was followed by a statistical method and is shown in Fig. 8 for the recrystallized plain carbon steel. The change in mean particle size is also included. In this figure  $N$  is the  $N$ th largest particle in a given volume of metal. During the first 10 000 cycles of fatigue, when the volume fraction is constant, true coarsening occurs, i.e. large particles grow at the expense of smaller ones. Upon further cycling, when the volume fraction decreases, the process seems to reverse itself, with large particles dissolving and smaller ones growing. Some of the very small particles which had dissolved completely during the initial enhanced coarsening, are even renucleated in the supersaturated matrix. The dissolution of the larger particles and growth of the smaller ones, soon leads to a form of dynamic equilibrium which is maintained until fracture of the specimen at about 120 000 cycles.

Quantitative calculations showed that enhanced diffusivity due to an excess vacancy production during fatigue, under these experimental conditions, can only account for an enhancement of 19. The only other alternative, an enhancement caused by dislocation movement, was therefore examined more closely. From the strain per cycle and the measured dislocation density it was calculated that in the early stages of fatigue, dislocations move over distances at least one order of

magnitude greater than the mean inter-particle spacing of carbides. A dislocation will thus 'visit' many particles of varying size during the initial period of fatigue. This could lead to rapid enhanced coarsening by the capture of solute at the smaller particles and transporting it to larger particles. Although dislocations move to and fro through the matrix during fatigue, the quantitative enhancement in coarsening rate should be similar to that where dislocations move unidirectionally, on condition that they move to and fro over distances greater than the inter particle spacing. The enhancements in coarsening rates of 3 000 found, during high temperature tensile testing of the same steels<sup>13</sup>, are thus of the same order as found here.

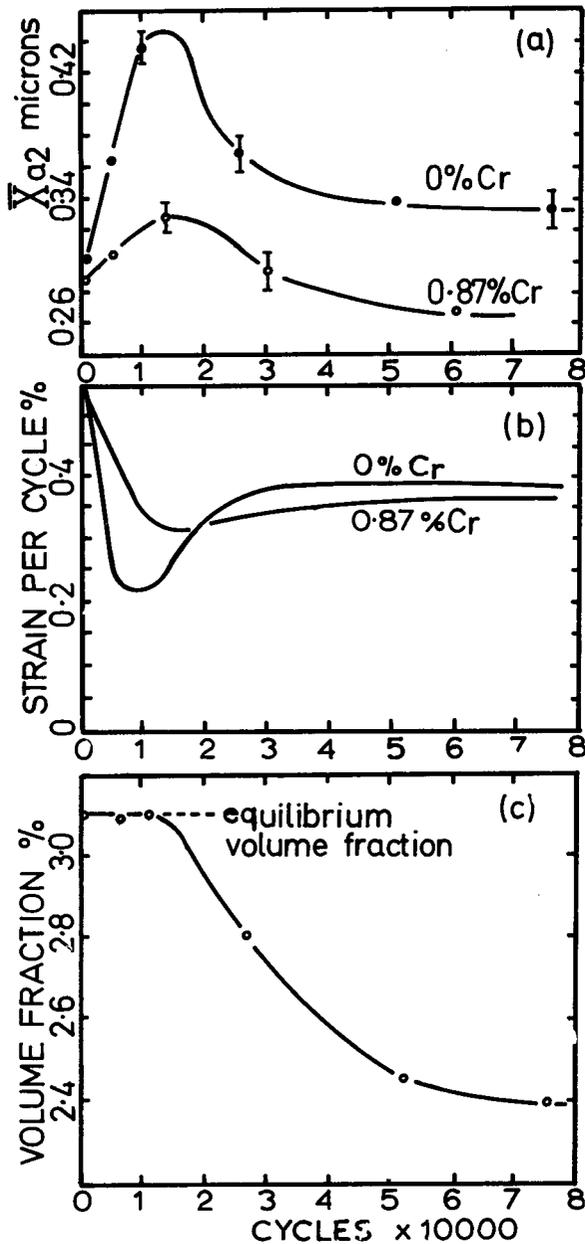


Fig. 7—Recrystallized steels, fatigued at 700°C, 10 500 lb/in<sup>2</sup> and 45 cycle/sec.

- (a) change in mean arithmetic volume particle diameter,
- (b) change in strain per cycle and
- (c) change in volume fraction of carbides for plain carbon steel only.

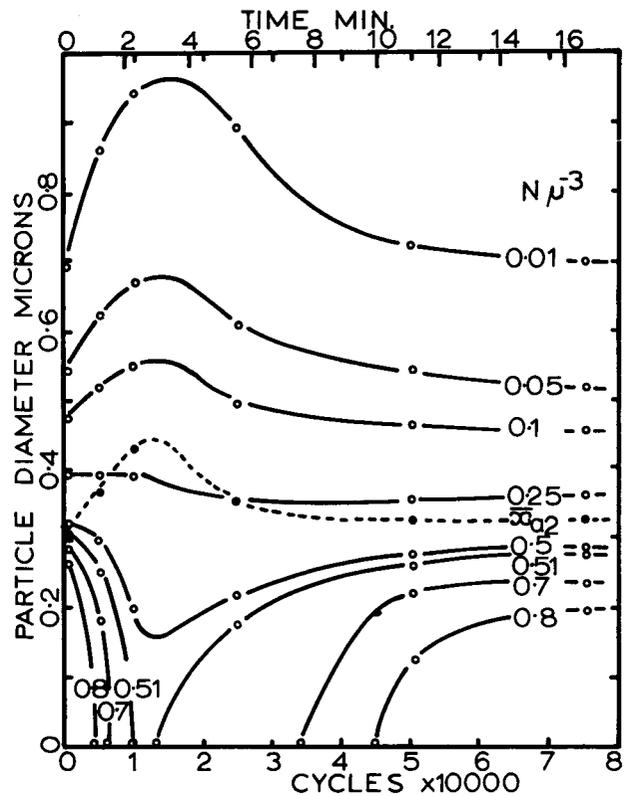


Fig. 8—Growth and dissolution of individually sized particles during fatigue of a recrystallized 0.21% plain carbon steel at 10 500 lb/in<sup>2</sup>, 700°C and 45 cycle/sec.

During the early stages of fatigue, work hardening, and therefore dislocation multiplication, takes place until a stage is reached (after about 10 000 cycles in Fig. 7 (b)) where dislocations move over distances less than the interparticle spacing. In the recrystallized plain carbon steel after 75 000 cycles at 10 500 lb/in<sup>2</sup> it was calculated that dislocations move over distances of about 0.6 microns during each fatigue cycle. This is far less than the mean interparticle spacing of 2.4 microns. Dislocations alternate, therefore, only between particle surfaces and the immediate surrounding matrix. Transport of solute from one particle to another by moving dislocations can no longer occur and strain enhanced coarsening becomes insignificant. Dissolution of particles could now be possible by an elastic interaction between the stress field of a dislocation and the solute atoms in a particle. Such an elastic interaction can lead to very high rates of stress-directed diffusion of solute away from the particle<sup>14</sup>. Dissolution can thus occur by the repeated 'draining off' of solute from the particles as the dislocations alternate between the matrix and a particle surface. An apparent supersaturation of the matrix can occur if these saturated dislocations are annihilated or are able to free themselves from the solute by nucleation of submicroscopic precipitates in the matrix. This will proceed until a dynamic equilibrium is established when the flux of solute back to the particles equals the stress-directed flux of solute, on the dislocations, away from the particles and macroscopic dissolution will cease. At this stage the diffusion distance of carbon for one half of a fatigue cycle, must be of the same order as half the interparticle spacing. This was calculated as 1.8 microns which agrees very well with

half the interparticle spacing of 1.2 microns for the recrystallized plain carbon steel after 75 000 cycles of fatigue.

From these observations it seems that the behaviour of carbides undergoing fatigue deformation must depend sensitively on the dislocation behaviour in the matrix. A more detailed investigation into the dislocation multiplication during early fatigue and the later dislocation behaviour is, therefore, desirable before the proposed mechanisms of enhancement and subsequent dissolution of the carbides during fatigue, can be proved conclusively.

### CONCLUSION

Significant effects, due to prior or concurrent deformation, have been found to occur in the coarsening process of carbides in the steels investigated. These effects are, however, expected to be smaller in intermetallic precipitation-hardened alloys, where only substitutional elements are involved, and may even be insignificant.

### ACKNOWLEDGEMENTS

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## Discussion

### Written Contribution

**C. E. Mavrocordatos** (Fellow): The authors of this paper — one of a series dealing with the mechanism of carbide particle growth with prolonged tempering at subcritical temperatures in plain carbon and chromium steels—must be congratulated on their excellent work.

The problem of the rate of growth of carbides at subcritical temperatures has a direct bearing on the machinability of alloy steels. It is an accepted fact, with ample theoretical justification, that the coarser the carbide particle, the softer the steel and therefore the easier to machine. One might venture here to suggest that as the size of the carbide particle increases, the easier it becomes for the chip to break off, thus satisfying yet another requirement of improved machinability.

The authors discuss in this paper not only the observed rate of growth of the carbide after a direct quenching treatment but also after cold deformation as well as after subjecting the steels to both creep and fatigue stressing conditions.

Their theoretical treatment of the observed phenomena has been clearly set out in their excellent paper.

One statement, however, appears to be rather contentious. In an effort to explain the difference in rate of growth of the carbide particle in the two conditions investigated (see Fig. 1), they attribute the greater rate of growth starting from the 'as-quenched' condition to

a greater initial density of dislocations, as compared with the specimens which were 'recrystallized' after cold-work.

Since the studies were carried out at temperatures much above the 550-600°C range, which is generally considered to be an adequate recrystallization temperature range for steels, one would expect that, at the coarsening temperatures they employed, a stable sub-grain structure would have ample opportunity to form soon in both conditions of treatment. Further, one would expect, that the initial differences in density of dislocations and vacancies would soon even out; and that the same state of equilibrium in both conditions will obtain after very little time at the elevated temperatures used.

Perhaps the authors will have an opportunity, at some future date, to present a further paper to this Institute in which this point might be cleared, and in which the hardness figures, which they reported in another paper,<sup>1</sup> could also be discussed and the reasons for some—perhaps apparent—discrepancies might be explained.

The contributor has carried out similar studies on a plain carbon steel, using various pretreatments as the initial condition. The results of this investigation will soon be published. In this investigation, also, different rates of growth were observed with different pretreatment, but the degree by which the rates differed were not so spectacular as the results, shown in Fig. 1, appear to be.

Dr Stumpf and Dr Sellars must be congratulated for their valuable work on this, as yet inadequately studied, subject of particle growth in a homogeneous matrix.

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**Dr G. T. van Rooyen\*** (Member): The low-carbon chromium steels which were used by the authors are representative of some of the types of steel which find application at medium high operating temperatures such as for boiler tubes in a steam plant. Boiler tubes are subjected to high pressure and highly superheated steam and the creep properties over a long period of time are therefore of considerable interest.

In creep tests, in which the long term creep properties of a material have to be evaluated, it is often not practical to duplicate the actual service conditions. The usual practice is to perform accelerated creep tests over a shorter period of time but at a higher temperature than that which the material will be subjected to in practice. Various extrapolation techniques are then used to predict the long term creep properties from the results of the accelerated tests. Often such extrapolations can be surprisingly accurate. Sometimes, however, it is found that the actual creep properties fall short of those predicted by the short term tests. In most instances the discrepancies have been traced to detrimental metallurgical structural changes which have been enhanced by the actual service conditions, but which have not manifested themselves during short term testing. Spheroidization, or the coarsening of carbides, is such a structural change which could give rise to creep rates in practice which may be higher than those anticipated; the study of the factors affecting such coarsening is therefore of great importance.

In general it can be said that the creep resistance diminishes very drastically when resolution or alternately agglomeration of precipitated particles begins. The creep properties are affected by the particle size, distribution and volume fraction of the second phase as well as by the coherency stresses between particles and matrix. In complex alloys precipitation usually occurs in service during creep and equilibrium phases are only attained after extended periods. As a matter of fact several metastable phases may precede the stable or equilibrium phases. If, during such a transition, large metal atoms have to diffuse through the lattice the equilibrium will be attained very slowly and systems that lead to complex compounds are usually more stable than binary compounds.

The coarsening of precipitates at high temperatures can be minimized in several ways such as:

- (a) The selection of precipitates which are crystallographically closely matched to the matrix and therefore remain coherent longer.
- (b) The use of dispersed phases which are practically insoluble in the matrix so that the solution of small particles and the consequent growth of large particles is slow.

The well-known high temperature alloys, such as Nimonic and Inconel for example, use  $\text{Ni}_3\text{Al}$  precipitated particles to restrain the creep rate. The  $\text{Ni}_3\text{Al}$  precipitates are isomorphous with the matrix and differ from the latter in lattice parameter by only 0.5 per cent. This mismatch is, however, enough to create appreciable strains around and inside the particle and in this way augment their effectiveness as barriers for creep deformation.

Inert phases, which are almost insoluble in the matrix and which are usually dispersed into the metal by means of powder metallurgy techniques, are particularly resistant to coarsening and are consequently effective up to very close to the melting point of the matrix. The best known application of this fairly recent development is the S.A.P. (Sintered Aluminium Powder) alloys in which alumina ( $\text{Al}_2\text{O}_3$ ) is dispersed into an aluminium matrix and T.D. Nickel alloys in which Thoria ( $\text{ThO}$ ) is dispersed into a nickel matrix.

The effect of dispersed particles on the steady state creep rate has been treated theoretically by Ansell and Weertman.<sup>1</sup> According to this theory mobile dislocations are prevented from moving freely through the metal lattice under the influence of the applied stress by the presence of second phase particles.

Creep deformation is then only possible when the dislocation can climb over the second phase particle. Climbing of a dislocation requires bulk diffusion and is therefore only possible at high temperatures. According to Ansell and Weertman the resulting creep rate at a particular temperature and applied stress is proportional to the square of the interparticle distance. The creep rate will, however, also decrease with an increase in diameter of the particle according to the equation.

$$\text{Creep rate} = \frac{C_1 \lambda^2}{d} \quad \text{where } C_1 = \text{material constant}$$

$\lambda$  = interparticle distance  
 $d$  = diameter of particle

If the volume fraction of the second phase remains constant, such as for example when coarsening of the particles are associated with a decrease in the total number of particles, the interparticle distance will increase as some of the particles grow larger. Under these conditions, then, the creep rate will be directly proportional to the interparticle distance.

Many papers have been published on the influence of recovery heat treatments and of superimposed cyclic stresses on the creep strength of alloys. Comparatively little, however, has appeared in the literature which relates the influence of these variables to the metallurgical structure and consequently to the interparticle spacing. In this respect the work reported by the authors is a great contribution to the knowledge of the subject.

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\*Head, Department of Metallurgical Engineering, University of Pretoria.