MECHANISMS OF DEFORMATION IN THE HOT WORKING OF NICKEL-BASE SUPERALLOYS

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A B S T R A C T

The mechanisms of hot deformation have been determined to be dynamic recovery and dynamic recrystallization which control the flow stress developed and the rate of crack propagation. Through their subsequent interaction with static recovery and static recrystallization, which occur whenever the metal remains hot after deformation, they also determine the structure and properties either upon cooling or at entrance to another stage of deformation.

As a preliminary step to understanding the deformation of precipitation hardenable nickel base superalloys; a review is made of the mechanisms observed in the simpler face centered cubic alloys of iron, cobalt and nickel. The hot working behavior of superalloys is reviewed and analyzed in terms of the mechanisms mentioned above.

INTRODUCTION

The nickel-base superalloys are designed for resistance to high temperature creep and oxidation. This resistance is achieved through solid solution with Cr, Fe, Co, Mo and W, precipitation within the grains of intermetallic phases γ', $Ni_3(AlTi)$, and ε, Ni_3Nb, and precipitation at the grain boundaries of discrete carbide particles MC, $M_{23}C_6$, M_6C, where M may be Ti, Nb, W, Mo or Cr. Alloys with better creep properties usually have increased quantities of solute and of intermetallic precipitates.

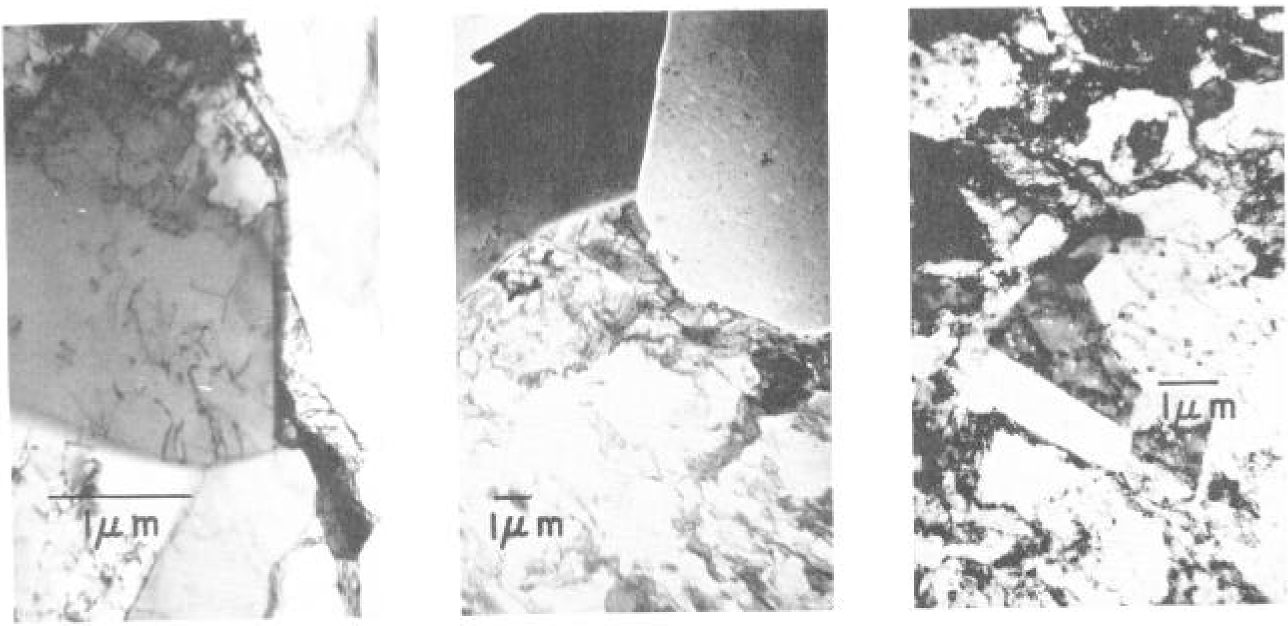
Since these alloys were designed to resist deformation at high temperatures, it is not surprising that they are very difficult to hot work; the ductility is limited and the flow stress is high. Furthermore, any addition in alloying which improves service qualities usually decreases the workability. Increased precipitates require an increased deformation temperature since the alloys are usually worked with the precipitates dissolved. The higher concentration of dissolved alloying elements (40-50% total) gives rise to higher flow stress, higher recrystallization temperature, and lower solidus temperature, thus narrowing the useful temperature range for hot forming. Because of the low thermal conductivity of these highly alloyed materials, contact with cold dies gives rise to severe temperature inhomogeneities (chilling) which diminishes the workability. Furthermore these alloys are subject during solidification to severe segregation and formation of columnar grains which diminish the ductility during primary reduction. The interdendritic regions of higher than normal concentration have a narrower working range than the homogeneous alloy. These deficiencies can be avoided by use of billets pressed from pre-alloyed powder. In the worked metal, bands of lower alloy concentration have less strengthening carbide and intermetallic precipitates, and larger grain size which comes from lower recrystallization temperature and more rapid grain growth. As a result the product suffers from lowered mechanical properties particularly in the transverse direction.

The objective of hot forming is to produce sound shaped parts of uniform composition and microstructure with grain size suited to subsequent solution, stabilizing and aging treatments. The practical procedures for the working of superalloys have been summarized by Sabroff, Henning and Boulger (1). The purpose of this paper is to reexamine the information about the hot working of superalloys in light of the current theories of deformation and rupture at high temperatures and strain rates. The summary of theory presents only the more important conclusions, omitting most of the supporting detail, because there have been published in the last few years, several reviews of the theories (2-6) and testing techniques (7-8) of hot workability.

The mechanisms of deformation at temperatures above half the melting point of the metal (^{o}K) and at strain rates between 10^{-3} and $2x10^{2}$ s^{-1} were deduced from microstructural changes and from the inter relationship between the mechanical parameters, which were observed in single phase metals and alloys. In general, the experimental deformations were conducted under conditions of constant and uniform strain rate and temperature. The mechanisms of deformation refer to those occurring during the actual forming and are entitled dynamic. Mechanisms which operate subsequent to the cessation of deformation and before the worked material is cooled down to room temperature are deemed static and are similar to the mechanisms which operate during the annealing of cold worked material. It is important to distinguish these two groups of mechanisms since they can be independently controlled to a certain extent. However, the starting point for the static processes is the microstructure at the end of deformation resulting from the dynamic mechanisms.

HOT WORKING - DYNAMIC RECOVERY

When aluminum is deformed in the hot working range to logarithmic strains between 0.5 and 3.4 and then cooled rapidly, the microstructure consists of the original grains considerably elongated, with a fine substructure and with scalloped boundaries (4 9-12). By means of transmission electron microscopy, the grains are seen to be divided by dislocation sub-boundaries into subgrains with diameters of the order of 2-10 μm (Fig. 1). The substructure resembles aluminum which has been coldworked and softened but not recrystallized by annealing; hence the name dynamic recovery. The subgrain size increases as the temperature of deformation increases or as the strain rate decreases:



(a) (b) (c)

FIG. 1 Hot worked microstructures as observed by transmission electron microscopy of thin foils. (a) Dynamic recovery substructure in 18%Cr-8%Ni austenitic steel which has been deformed in hot torsion at 1000°C (1832°F) and 0.68 s^{-1} to a strain of 1.11 (54). (b) Statically recrystallized nuclei are growing into the dynamically recovered substructure in Cu which has been rolled at 600°C (1112°F) and 20 s^{-1} to a strain of 2.2. The specimen was quenched in 0.1 s after leaving the rolls (17). (c) Substructure in dynamically recrystallized Cu which has been twisted at 800°C (1472°F) and 11.1 s^{-1} to a strain of approximately 30. The specimen was quenched during deformation to prevent static recrystallization (23).

$$d^{-1} = -0.6+0.8 \log Z \qquad (12)\text{(Eqn. 1)}$$

where d = subgrain diameter by ASTM analysis

Z = $\dot{\varepsilon} \exp (+Q/RT)$

= temperature compensated strain rate

$\dot{\varepsilon}$ = strain rate (s^{-1})

Q = activation energy of the mechanism, kcal/mol (kJ/mol)

R = universal gas constant (2 cal/mol ^{o}K) (8.3 J/mol^{o}K)

T = absolute temperature (^{o}K)

Furthermore as the subgrain diameter increases with decreasing Z, the dislocation density within the subgrains and in the sub-boundaries decreases; the sub-boundaries change from tangles to orderly arrays.

The stress strain curve of aluminum deformed at a constant strain rate and at a constant elevated temperature exhibits a horizontal regime above a strain of approximately 1 which varies inversely with Z. This regime, in which temperature, strain rate and stress do not vary as strain increases, is called steady state deformation and is equivalent to the second stage of creep. During this regime, the subgrain size and misorientation remain constant, thus keeping the dislocation density constant and giving rise to constant flow stress. It thus appears that the rate of annihilation of dislocations (recovery) is high enough to balance the rate of generation of dislocations. The annihilation of the dislocations in the subgrain walls leads to their repeated disruption; subsequently new walls form at an average spacing equal to the subgrain diameter so that the subgrains remain equiaxed, although the grains in which they are embedded become progressively elongated. During the initial hardening regime the dislocations accumulate until equilibrium is attained, this is equivalent to first stage creep. At higher Z, (lower T, higher $\dot{\varepsilon}$) the balance is reached at higher dislocation density since the rate of recovery is lower. The steady-state flow stress depends on the substructure that evolves under the conditions of strain rate and temperature (Fig. 2).

The steady rate flow stress is dependent on the temperature and strain rate of deformation. Of the several mathematical relationships that have been proposed, none have a strict theoretical foundation; a convenient one (Fig. 3) is:

$$f(\sigma) = A (\sinh \alpha\sigma)^{n'} = \dot{\varepsilon} \exp (Q/RT) = Z \qquad (11)\text{ (Eqn. 2)}$$

where σ = flow stress in steady state (or at a fixed strain in the hardening regime)

A, α, n', = material constants determined from the test data.
The activation energy, Q, is approximately equal to that for creep or for self diffusion, thus suggesting that the mechanism involves the diffusion of vacancies to climbing dislocations i.e. dynamic recovery. This equation applies not only to hot work but also to creep without any discontinuity (11). The totality of the evidence indicates the mechanisms of creep and hot working of aluminum are identical.

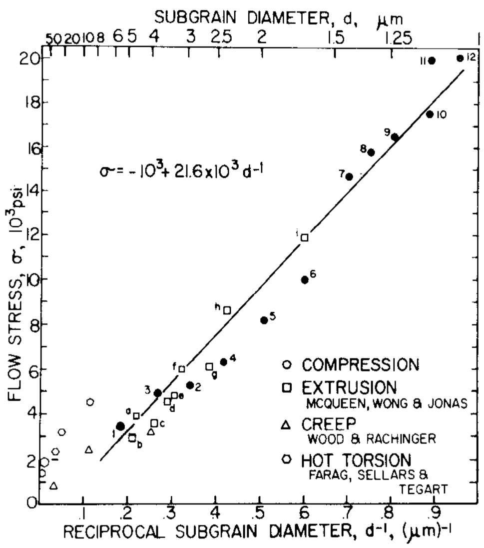
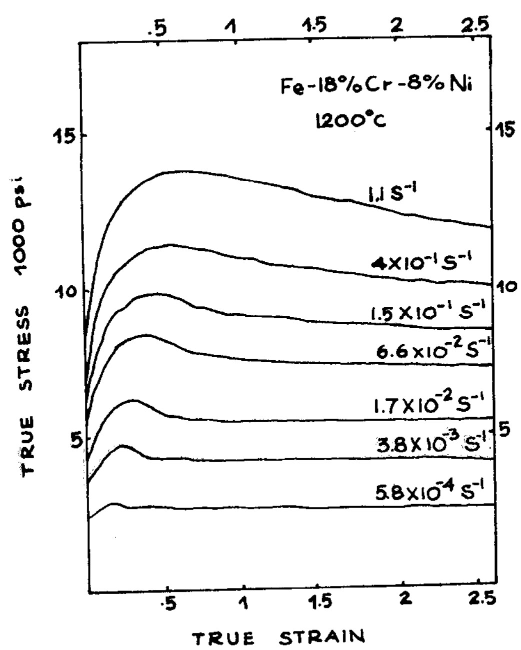
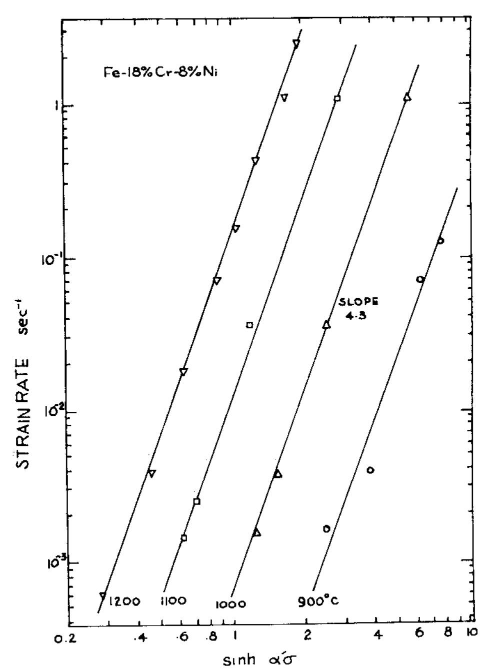


FIG. 2 The relationship between the steady state flow stress of aluminum and the equilibrium substructure which has developed during the deformation (12).



(a)



(b)

FIG. 3 Flow curves exhibiting steady state deformation in which the parameters can be correlated by the hyperbolic sine formula. (a) Effective stress-effective strain curves of austenitic stainless steel at 1200°C (2192°F) (36). The curves are characteristic of metals in which dynamic recrystallization begins at the peak. The continual decrease at high strain rates is the result of partial adiabatic heating. (b) The strain rate, temperature and steady state flow stress of austenitic stainless steel are correlated according to Eqn.2 with the constants

Q = 97 k cal/mol (405kJ/mol) $\qquad \alpha = 9.27\times10^{-5}\ (\text{psi})^{-1}$ $\qquad$ and $\qquad n' = 4.30$

Dynamic recovery appears to be the controlling mechanism of deformation in aluminum. There is, of course, considerable grain boundary sliding but this represents less than one per cent of the strain at hot working rates; its role in rupture will be presented later. Recrystallization during the deformation has never been observed. Moreover the substructure indicates that it is unlikely to occur because dynamic recovery maintains the sub-boundaries at a sufficiently low misorientation that they never become capable of migrating. The substructure seems to be of a scale that prevents the original grain boundaries from bulging into new grains.

After deformation is stopped, the hot worked aluminum can recrystallize if held at the working temperature or if cooled very slowly. If held at the working temperature, the rate of recrystallization is higher, the higher the temperature of working. If a common heat treating temperature is employed after working, the rate of recrystallization is greater the lower the temperature of working since the dislocation density is higher. It is possible, even under industrial conditions, to cool the product sufficiently rapidly to prevent recrystallization and retain the dislocation structure. The flow stress at room temperature varies with the hotwork subgrain diameter:

$$\sigma_y = \sigma_o + 11d^{-1.25} \qquad (13\ 14)(\text{Eqn. } 3)$$

The substructure can be retained in non-heat-treatable alloys to improve the strength and in Al-Cu precipitation hardening alloys to improve the uniformity of precipitate and hence the aged strength. Retention of a subgrain structure improves creep resistance over a limited temperature range (15 16).

During hot working all metals undergo dynamic recovery; however, the mechanism may not be as effective as it is in aluminum so that other mechanisms may come to predominate at larger strains. The body-centered cubic metals, α-Fe, Mo, and W, seem to be very similar to aluminum in their mechanisms and microstructural development. It is other face-centered cubic alloys, such as nickel, copper, and brass, that behave quite differently; the difference becomes greater with decrease in the stacking fault energy, which controls the rate of recovery though its effect on dislocation climb and crossglide. With decreasing stacking fault energy for deformations at the same fraction of the melting temperature (^oK), the subgrains became smaller and the dislocations in the sub-boundaries became more densely packed and more tangled (17). Because of the greater misorientation between the subgrains, a new mechanism, dynamic recrystallization starts during deformation. The strain at which it starts, is less than that for steady-state dynamic recovery and increases as Z increases. Under the usual conditions of temperature and strain rate employed in industrial operations, the critical strain is sufficiently high that dynamic recrystallization does not start in operations such as forging or rolling.

For the metals of low stacking fault energy, because of the very dense substructure, static recrystallization occurs very rapidly when the deformation is stopped, so that it is very difficult to retain the as-worked structure (Fig. 1). It is possible by controlling the strain rate, the temperature of deformation, the strain per pass, the time between passes, and the cooling rate, to produce a suitable grain size (5 18-20). Such procedures are used in the controlled rolling of plate and in hot-cold work schedules for forging.

HOT WORKING - DYNAMIC RECRYSTALLIZATION

The stress-strain curves for Cu and Ni exhibit a peak which is followed by a decline in flow stress (Fig. 3). The decrease in flow stress is due to the first wave of recrystallization passing through the specimen (4 21 22). If Z is low the flow curve becomes a sinusoidal line in which each peak is the start of a fresh wave of recrystallization and each valley marks the completion of the wave. If Z is high, the strain for completion of a wave of recrystallization overlaps with the strain for nucleation in the material which recrystallized early in the previous stage. The result is a steady state deformation regime in which the flow stress is constant, is lower than the peak stress and is much lower than the steady-state flow stress which would have resulted from dynamic recovery alone. During steady state, the work piece contains a spatially uniform distribution of grains which have different flow stresses dependent on the strains they have undergone since their recrystallization. In the electron microscope specimens which have been quenched extremely rapidly, exhibit small nuclei completely free of dislocations and grains with various densities of substructure (Fig. 1)(23). In the optical microscope, the grains remain equiaxed and similar in size regardless of strain. The grains are large for deformation at lower Z, i.e. lower strain rate and higher temperature.

The dependence of the steady state flow stress on the deformation temperature and strain rate can be expressed by the same equation as that for dynamic recovery (Eqn.2, Fig. 3). However, the activation energy is usually quite different from that for creep or self diffusion and cannot easily be compared with that for static recrystallization because of the variability with even small changes in composition.

After the deformation is halted, recrystallization occurs very rapidly and may occur in two stages which are distinguishable only in certain cases (24 25). The first is meta-dynamic static recrystallization which has no incubation period since it occurs by the continued growth of the dynamic nuclei formed just at the end of deformation. The other is ordinary static recrystallization for which nuclei form in the normal way after the deformation has ceased. The final static recrystallized grain size can be selected by controlling strain rate, temperature, strain per pass, interval between passes and rate of cooling. Industrially there may be little difference in the final product whether the mechanism was solely dynamic recovery or also dynamic recrystallization.

RUPTURE AT HIGH TEMPERATURES AND HIGH STRAIN RATES

The initiation and propagation of wedge-shaped grain boundary cracks has been studied in face centered-cubic alloys with limited ability to recover (35 26-31). The cracks initiated at triple points or ledges as a result of grain boundary sliding which, although only a small fraction of the total strain (32), is still large in comparison with the amount occuring in a creep test. When dynamic recrystallization does not take place, either because the temperature is near the bottom of the hot working range or the strain rate is low, the cracks grow quite rapidly

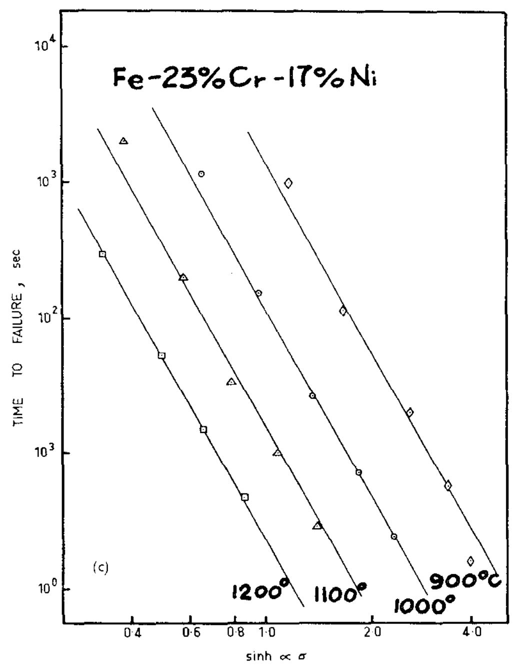


FIG. 4 The time to rupture can be correlated with the deformation conditions by Eqn. 5. This data is taken from hot torsion tests on fully austenitic stainless steel Fe-23%Cr-17%Ni (5).

along stationary boundaries and bring about a low ductility. At higher temperatures, the cracks initiate more frequently but propagate very slowly because the boundaries migrate away from the cracks during dynamic recrystallization. A crack can propagate only when it captures a boundary and then it is only for a short period of time. Under these conditions the ductility is very high and is many times greater than that when dynamic recrystallization is absent. If static recrystallization takes place between stages of deformation then the cracks initiated in the first stage are isolated from the grain boundaries and cannot propagate during the second stage. The most satisfactory criterion for predicting failure on the basis of the deformation conditions appears to be the following equations in which t_r is the time to fracture (Fig. 4)(5 28 29 31):

$$t_r \dot{\varepsilon} = \text{constant} \qquad \text{(Eqn. 4)}$$

and with substitution from Eqn. 2 for $\dot{\varepsilon}$

$$t_r = A' \, (\sinh \alpha\sigma)^{-n'} \exp\,(Q/RT) \qquad \text{(Eqn. 5)}$$

The experimentally measured values of n' and Q may be the same as for the experimentally measured strength - strain correlation of Eqn. 2.

The ductility of bcc metals and fcc metals with high stacking fault energy which deform solely by dynamic recovery seems to be higher than those undergoing dynamic recrystallization (3 26 27). It would appear that the formation of a scalloped boundary reduces grain boundary sliding so that cracks do not have an opportunity to grow.

Inclusions or second phase particles which are brittle or much stronger than the matrix and are larger than about 10μm serve as crack initiators and can assist crack propagation by preventing grain boundary migration (3 33-35). The precipitates are most damaging if they are at the grain boundaries in a massive or continuous form. The melting of the alloy at segregated grain boundaries or of a second phase causes the metal to crumble and determines the upper limit of the hot working range.

HOT WORKING OF FACE-CENTERED CUBIC ALLOYS OF TRANSITION METALS

When heat treated for service, the nickel base superalloys constitute a distinct class of alloys because of the corrosion - resistant concentrated-solid-solution matrix and the precipitates of the intermetallic $Ni_3(TiAl)$. However in the condition in which they are usually hot worked ($Ni_3(TiAl)$ in solution) they are a member of a series of face-centered cubic alloys which may be based on iron, cobalt or nickel. Whereas the lower concentration alloys of those three elements exhibit several distinct characteristics, the higher concentration alloys share many similarities because of alloying with each other and with common solutes such as Cr. It is useful to progress from the simpler alloys to the more complex.

Plain Carbon and Low Alloy Steels

The plain carbon and low alloy steels are characterized by a substantial dissolved carbon content which lowers the austenitizing temperature without significantly lowering the temperature for δ formation or for melting. The small quantities of alloying elements which were added for improved hardenability or hardness are in solution. The stress strain curves of Rossard (36), which exhibit a peak followed by steady state or oscillations at lower strain rates, indicate dynamic recrystallization at high strain. The relations between the flow parameters in steady state follow Eqn. 2 with activation energy varying from 77-110 kcal/mol (322-460 KJ/mol) as carbon content rises. By means of special metallographic techniques, which expose the prior austenite grain boundaries it has been observed that at a strain up to approximately 50% reduction, the mechanism is dynamic recovery which is followed by rapid static recrystallization (18 19 37-39). The strengthening effect of the dynamically recovered substructure has been exploited in the ausforming process. Refinement of the austenite grain size can be achieved by controlling reduction per pass and time between passes in the rolling mill (18-20 40). The ferrite grain size can be made equal to or larger than austenite grain size by controlling the rate of cooling.

The kinetics of static recrystallization have been examined by means of interrupted mechanical tests. Wilbur, Capeletti and Childs (41 42) observed that the rate of recrystallization increased with amount and temperature of deformation so that it was completed in a second at 1015°C (1900°F). Softening by recovery was observed prior to recrystallization. Djaic and Jonas (24 25) have extended the technique to show that meta-dynamic static recrystallization has no incubation period and may be followed by a distinct stage of static recrystallization.

The effect of NbC precipitates which are uniformly distributed and sufficiently fine that they do not initiate cracks have been examined (18-20). The particles raise the flow stress presumably by limiting recovery and stabilizing the substructure as has been observed in creep (42 43). By preventing grain boundary migration, the particles decrease the rate of static recrystallization by two orders of magnitude compared to plain carbon steels. This makes possible the production of an ultra-fine austenite grain size which transforms to stronger, tougher ferritic-pearlitic structures. If the austenite is allowed to transform to ferrite before it recrystallizes, the finest ferrite grain size is approximately equal to the shortest dimension of the austenite grains; thus, the refinement is dependent on the reduction and the initial austenite grain size. It would appear that these precipitates prevent dynamic recrystallization at high strains.

As the concentration of dissolved alloying elements increases, the hot strength increases by 25 to 50% (1 45), and the ductility diminishes by as much as 50% (Ni and Mn in small quantities increase ductility). The ductility usually increases with increased rate of deformation but this may only be the result of adiabatic heating. Inclusions play a prominent role in initiating fracture. Robbins, Shepard and Sherby (33 34) have shown that, when the steel cools into the α/γ two-phase region, the formation of α lowers the ductility substantially (35).

Cobalt

Hot torsion stress-strain curves of face centered cubic cobalt by Jacquerie and Habraken (46) have the peak and steady state regime characteristic of dynamic recrystallization.

Nickel

In torsion tests of nickel (21 29 47-49), the characteristic stress-strain curve exhibits a peak followed either by a steady state regime or at low strain rates by oscillations. Luton and Sellars (47) correlated the strength, strain rate and temperature by Eqn. 2. The activation energy for pure nickel was found to be 56 kcal/mol (234 KJ/mol) which is considerably lower than that for self diffusion. These results in association with the following microstructural evidence are indicative of dynamic recrystallization. Specimens rapidly cooled from the steady state region appear recrystallized but contain a dislocation substructure. In some cases, nucleation occurred by grain boundary bulging. Specimens deformed less than the peak strain exhibit a subgrain structure (17) and an incubation time for the start of recrystallization but there is none for specimens deformed into steady state deformation.

Sah, Richardson and Sellars (40) measured the incubation time and critical strain for dynamic recrystallization as a function of strain rate at 880°C(1616°F). As the rate of strain increased, recrystallization started at shorter times but after greater critical strains. From the observed rate of recrystallization, the rate of strain hardening (stress-strain curve before the peak) and the critical strain, it was possible to make a calculation of the shape of the stress strain curve after the peak which agreed with the experimental data. At high strain rates and high temperatures the time to reach the critical strain at any point is less than the time for completion of recrystallization in the region, some points will begin to recrystallize a second time before the entire region has recrystallized, thus the dynamic recrystallization occurs continually. On the other hand, at low strain rates, the time to reach the critical strain at any point is much greater than the time for recrystallization of the whole region with the result that the dynamic recrystallization is periodic and the flow stress oscillates.

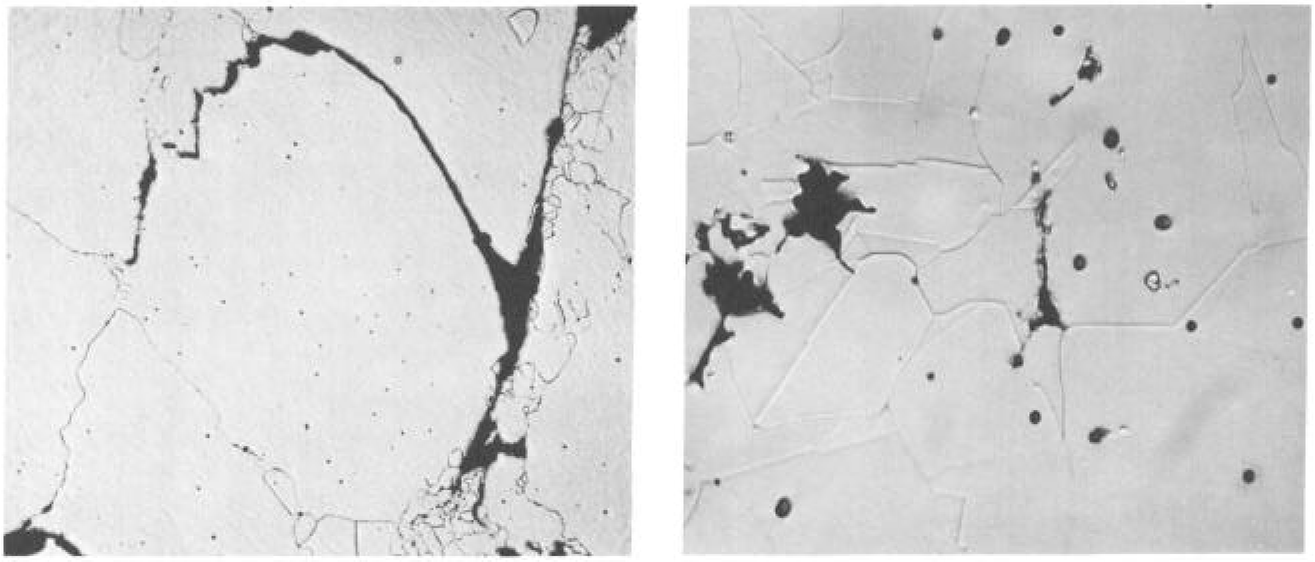
The time to fracture was related by Luton and Tegart (31) to the conditions of deformation by Eqn. 5. In Fig. 5, grain boundary cracks were halted as the boundaries were removed by dynamic recrystallization. The isolated cracks often became amoeba like voids. Fracture occurred by the linking up of the voids. Shapiro and Dieter (29) divide the rupture behaviour of nickel into three temperature ranges; in all cases the cracking starts at the peak stress. In the low temperature range the metal strain hardens up to a fairly high strain and fractures at the peak stress by transgranular shear. In the intermediate range in which there is no dynamic recrystallization, wedge-shaped cracks form at grain boundaries and extend rapidly to give very low ductility with little extension after the peak. In the high temperature range in which dynamic recrystallization takes place, the grain boundaries migrate away from any cracks that form thus preventing them from propagating. After the peak stress, the flow stress declines to the steady state level and high strains are possible before ultimate fracture. At low strain rates where the recrystallization is periodic, the boundaries remain stationary for longer periods so that a much more extensive crack network forms and there is lower ductility than at high strain rates. The temperature for transition from intermediate to high ranges and the strain for the maximum stress increase as the strain rate is raised.

Luton, Sellars and Tegart (31 47) also studied the behaviour of Ni-Fe alloys up to 20% Fe and found it to be similar to that of pure nickel. The stress at the peak strain and in steady state increased as the Fe content increased; however, this effect diminished as the temperature increased to $0.9T_m$. For strains less than the peak, the incubation time for static recrystallization increases with iron content. The interdependence of the flow parameters obeyed Eqn. 2 with activation energies of 71 kcal/mol(298 kJ/mol) for 5% Fe, 81(339) for 10% Fe, and 94(393) for 20% Fe. Between 762°C (1403°F) and 1279°C (2334°F), the dynamically recrystallized grain size during steady state decreases as the flow stress increases according to the equation.

$$\sigma_{ss} = K\, d_G^{-3/4} \qquad \text{(Eqn. 6)}$$

where K decreases from 1.45 for pure Ni to 0.82 for 20% Fe. This indicates that for a given flow stress the grain size of the alloys is lower as the Fe increases because the dissolved Fe decreases the rate of boundary migration. The flow stress must be controlled by the dislocation substructure rather than by the grain size.

Alloys of Fe-25%Ni have been studied by White and Rossard (30). Metallographic examination confirmed dynamic recrystallization was taking place during the steady state



(a) (b)

FIG. 5 Grain boundary cracking in Ni-5%Fe deformed at a strain rate of $2\times10^{-3}\ s^{-1}$. (a) At 760°C (1400°F) the cracks propagate (ε=0.35). × 125. (b) At 934°C (1710°F) dynamic recrystallization has prevented propagation (ε=1.6). × 625. (31).

deformation which followed the peak in the stress-strain curve. Crack propagation was inhibited and ductility augmented whenever dynamic recrystallization took place or whenever static recrystallization occurred during the intervals between passes.

Austenitic Stainless Steels

The results presented refer to the most widely studied steel of this type which is Fe-18%Cr-8%Ni; only significant differences for other alloys will be mentioned. Rossard (36) determined the stress strain curves over a range of temperature and strain rate showing that the curves had the characteristic shape for dynamic recrystallization (Fig. 2)(50 51). The flow parameters could be correlated by Eqn. 2 (Q=97 kcal/mol)(405 kJ/mol) and the flow stresses were approximately twice those of carbon steels as a result of the high rate of strain hardening (1 36). In general, the hot strength was greater and the ductility lower when the starting grain size was larger and the amount of Cr, Mo or W increased (1 45 52). After deformation in the steady state regime, the grain size was finer for higher Z, and static recrystallization would occur upon holding at temperature.

At lower strains, as in creep (32), a subgrain structure developed (Fig. 1) (36 50-56); the size was smaller for lower temperature and greater strain rate and strain (57). When this substructure is maintained to room temperature, the strength is increased about 50%, the effect is easier to achieve and is greater if the steel contains Nb (58). A substructure was observed in steel containing $Ni_3(AlTi)$ only when worked above the solution temperature (56).

Metal which has been worked in the dynamic recovery range will recrystallize when maintained at temperature; the rate being greater when the temperature is higher, the reduction greater and the initial grain size smaller (50 52 59). These results were confirmed by more precise measurement of the kinetics of recrystallization by Capeletti et al (42) using interrupted tensile tests and by Buhler, Bobbert and Rose (53) who used interrupted tensile tests, metallography and changes in preferred orientation (for Fe-18%Cr-11%Ni).

Polygonization during deformation was observed in Fe-36%Ni-10%Cr by Tamhankar, Plateau and Crussard (26 27). Fracture occurred by intergranular cracking since the grain boundaries did not migrate being pinned by precipitate particles.

The ductility of fully austenitic Fe-23%Cr-17%Ni was studied over a range of temperatures (5). The fracture strain, temperature and stress could be correlated by Eqn. 5 with $n' = 4.9$ and $Q = 80$ kcal/mol (335 kJ/mol) which differ from the constants for strength data $n' = 5.3$, $Q = 96$ kcal/mol (402 kJ/mol). This alloy shows increasing ductility with increasing strain rate. On the other hand Fe-26%Cr-10%Ni which is 10% δ ferrite has the same values of n' and Q for both fracture and strength correlations. Results for Fe-23%Cr-17%Ni appear in Fig. 4 .

The presence of δ ferrite in small quantities always lowers the ductility of austenite (1 5). For Fe-12%Cr examination of the ductility as a function of temperature showed that minima occur at $1025^{\circ}C$ ($1877^{\circ}F$) and at $1300^{\circ}C$ ($2372^{\circ}F$) due to two-phase regions of α/γ and γ/δ respectively (52). The temperature range of single phase austenite narrows as the chromium increases and the carbon decreases. In alloys containing δ ferrite minimum ductility occurs when the δ ferrite content is about 30%. Although the ductility rises with increasing percentage of δ ferrite, the possibility of over heating and melting increases (1). Ferrite may also be present as a result of segregation during casting. In alloys such as Fe-25%Cr-6%Ni, mixtures of ferrite and austenite can exhibit superplastic properties when they are in the microduplex structure (60). This structure, which is produced by hot rolling, consists of ferrite and austenite grains about 3μm in diameter which are uniformly blended. Brittleness of the two phase structure can arise from the formation of chromium carbide at the interphase boundaries. These carbides can be avoided by lowering the carbon content or by adding Ti to form matrix carbides.

HOT WORKING OF SUPERALLOYS

Analysis of the data reviewed in the previous section shows a consistent behavior which is not affected substantially by major changes in composition. Since the compositions and microstructures of the superalloys are not greatly different from the above alloys under hot working conditions one could expect to find the same mechanisms. At the commencement of deformation (up to 50-80% reduction) the mechanism is dynamic recovery and subgrains form. If the deformation is continued to high strains, dynamic recrystallization will take place with the formation of equiaxed grains containing a variable substructure. Static recrystallization can take place after either mechanism if the working temperature is maintained. With increased solid solution alloying and in the presence of fine precipitates, the recrystallization either dynamic or static is delayed and slowed down. Grain boundary cracking is the usual mechanism of fracture; however its progress can be retarded and the ductility increased by the commencement of dynamic recrystallization. Coarse precipitates hasten fracture.

Review of the literature on the hot working of superalloys reveals that much of it is devoted to practical improvements in processing to achieve higher yields of more uniform and consistent product. The information concerning the mechanisms that can be gleaned from these publications will be summarized. There are only a few papers which have discussed the mechanisms to any extent.

The initial deformation causes strain hardening (61-63) and results in a structure which is not recrystallized (62 64-71) and which contains subgrains (72-75). The flow stress is of the order of 3 times that of low alloy steels and the rate of strain hardening is as high as that of an austenitic stainless steel (1). It is easier to observe the as-worked structure in cases where the deformation is below the ε or γ' solvus since the precipitates retard recrystallization (66 68 74 75). In Fig. 6, Oblak, Owczarski and Duvall (74) show that the substructure of Udimet 700 is stabilized by the presence of the precipitates as has been observed in TD Nickel (42) or Fe-18%Cr-10%Ni containing $Cr_{23}C_6$ (43). Because of the smaller amount of γ' during hot working, the substructure is quite different from that observed in aged metal after creep at lower temperatures (76). Retention of the hot worked substructure can improve the creep resistance at lower temperatures (73 75). Static recrystallization will result if cooling is delayed; the grain size is finer for deformation at lower temperatures, higher strain rates and greater reductions (1 68 70 77). Weiss, Grotke and Stickler (72) have observed static recrystallization nuclei growing into the hot-work substructure in Inconel 600.

Oblak and Owczarski (75) have developed the possibility of retaining the polygonized hot-worked substructure into a thermo-mechanical treatment for Udimet 700. Prior to hot working there is a 4 hr. solution anneal at 1171°C (2140°F) and a 4 hr. preliminary aging at 1066°C (1950°F) to precipitate the γ'. After working to a strain of 1.50 (78% reduction) there follows a further aging at 843°C (1550°F) for 4 hours and at 760°C (1400°F) for 16 hrs. The treatment greatly improves the room temperature mechanical properties and the creep resistance up to 760°C (1400°F). A treatment involving 60% reduction at 1010°C (1850°F) did give slightly inferior creep resistance to standard Udimet 700 at 704°C (1300°F) and above. In a thermomechanical treatment for Astroloy, mill annealed alloy was rolled to a 20% reduction at 843°C (1550°F). This refined the precipitate morphology giving better room temperature properties but poorer creep resistance (67).

When the deformation is continued to high strains, the flow stress passes through a maximum and then decreases to a steady state regime. Shapiro, Muller and Dieter (61 62) have deformed Inconel 600 in torsion and have been able to observe the incidence of dynamic recrystallization after the peak. In general dynamic recrystallization is promoted by higher temperatures and higher strain rates. In hot torsion tests of Udimet 700 between 1060 and 1142°C (1940-2090°F), Young and Sherby (63)(Fig. 7) observed that the grains remain equiaxed and constant in size during steady state which is indicative of dynamic recrystallization. The microstructures observed after torsion were almost identical to those observed after extrusion at the same temperature, strain rate and strain. At higher working temperatures, the grain size was larger (62 63) and the γ' precipitate particles were fewer and larger but did not seem to have coalesced compared to undeformed specimens given the same thermal treatment. The occurrence of dynamic recrystallization in the presence of the γ' precipitate is somewhat surprising because Udimet 700 has been observed to resist recrystallization after rolling to a strain of 1.5 at 1065°C(1950°F)(74-75); however, in the latter case, the precipitates were finer, more numerous and more iniformly distributed.

The ductility is greatly improved if inclusions are eliminated, segregation reduced and the grain size reduced (1 5) Overheating before deformation can cause grain growth and increased dissolved alloy content which considerably reduces the ductility (70 72 77). Ductility of Udimet 700 was higher with the γ' precipitate than without because there was considerably less grain boundary sliding and cracking (74). The incidence of dynamic recrystallization inhibited propagation of grain boundary cracks and greatly raised the ductility of Inconel 600 in the manner described previously for simple nickel alloys (61 62).

Bailey (70 71) has conducted an extensive series of Gleeble tensile tests on Waspalloy, AL 718, and Udimet 625; similar tests have been carried out on Inconel 600 (72). In these tests as the temperature is raised, the ductility, measured by percent reduction in area at the neck, rises from a low value to a maximum and then decreases sharply as complete intergranular fracture occurs. The fracture at low temperatures (Udimet 700, 816-927°C (1500-1700°F); Waspalloy, 982°C (1800°F); and AL 718, 871°C (1600°F)) is partially intergranular and the worked material is at most slightly recrystallized. As the temperature rises, there is evidence of more recrystallization and the fracture mode becomes transgranular with considerable improvement in ductility. In Udimet 625, high ductility at 760°C (1400°F) and a transgranular fracture are associated with almost complete recrystallization. Since the inhibition of intergranular fracture in the intermediate temperature range is associated with observable recrystallization as in the hot torsion of Ni, Ni-Fe alloys, austenitic stainless steel and Inconel 600, it is concluded that in the Gleeble tests the recrystallization is dynamic and inhibits the propagation of grain boundary cracks. However, although optical metallography exposed the recrystallized grains, there was no transmission electron microscopy which could conclusively determine whether the new grains had been deformed or not; i.e., whether the recrystallization was dynamic or static. If the new grains are statically recrystallized, a new explanation for the inhibition of grain boundary fracture is needed.

The review of the literature shows that the mechanisms of dynamic recovery and recrystallization can account for the phenomena observed during hot forming of superalloys. However, it also is apparent that the processing of the age-hardenable superalloys involves many other steps subsequent to forming such as solution and precipitation of strengthening particles. Products with satisfactory properties can be manufactured only if attention is given to specifying each treatment as a part of the comprehensive procedure.

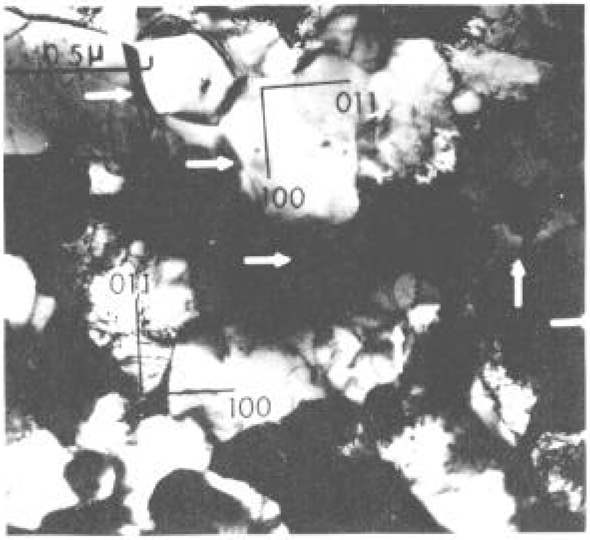


FIG. 6 This transmission electron micrograph illustrates the substructure present in thermomechanically treated Udimet 700. The specimen was aged to precipitate γ′ and was swaged at 1950°F to a reduction of 78% (74).

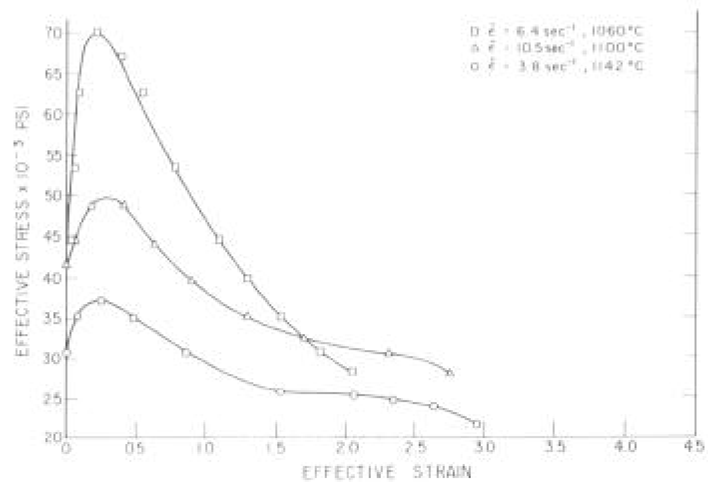


FIG. 7 Effective stress-effective strain curves from hot torsion tests on Udimet 700. The tests were not continued to fracture but interrupted at the strains shown so that the microstructures could be compared with those of extrusions produced under the same conditions. The rise in flow stress is the result of hardening during dynamic recovery and the drop is the result of dynamic recrystallization (63).

HOT COMPACTION OF SUPERALLOY POWDERS

The production of superalloy billets by compaction of prealloyed powders is important as a means of improving the workability by eliminating the segregation and columnar grain structure which is commonly found in cast ingots. It would appear to make possible the forming of certain alloys which after the most careful industrial casting practice cannot be worked at all. Satisfactory billets require prealloyed powder of precise composition, uncontaminated by oxidation and of suitable shape, size and size distribution. Facilities must be available for hot compacting the powder into billets without contamination from the environment. Cold pressing is unsatisfactory (78) because the high strength of the powder leads to such inadequate cold welding that the green strength is very low and limits the densification such that the shrinkage during sintering is unacceptable. Sintering without pressure which relies on surface and volume diffusion to enlarge the welds between the particles and to transport material into the voids (79 80 81) is too slow, and may result in non-uniform densification and poor dimensional control. What is needed is compaction at a temperature where the alloy has a much lower yield stress.

Hot pressing of the powder in a die (69) depends on several mechanisms. First, rapid deformation as in hot working and pressure welding when the force is initially applied. Second, a combination of dislocation creep and volume diffusion driven by both the applied stress (Nabarro-Herring microcreep) and by the surface energy (Kuczynski sintering) (69 80, 81). These latter mechanisms are very slow and tie up valuable press capacity. The dies are costly to prepare and maintain and present difficulties related to atmospheric control and dimensional retention. The compaction technique which gives satisfactory economic results is sealing the powder in an evacuated can succeeded by a normal hot forming process such as forging, extrusion or rolling (82-85). The mechanisms operative in this process are then pressure welding and the hot working mechanisms described earlier. The process has problems in prevention of contamination from the protective container and in removal of it.

Hot compaction has been shown to be technically successful. Billets and preforms have been produced which are free of contamination, possess much higher workability and exhibit considerably improved creep resistance (69 82-85). As a result of the very fine particle size achieved, superplastic deformation of 1N 100 and Udimet 700 has been achieved with high ductility and reduced forming stress(84 85). To give the product satisfactory creep resistance, a 56 hour grain-coarsening treatment at 1245°C (2270°F) was necessary.

C O N C L U S I O N S

The hot working mechanisms of dynamic recovery and dynamic recrystallization function in the nickel-base superalloys as they do in the simple face-centered cubic alloys of iron, cobalt and nickel. The hot-worked substructure can give rise to subsequent static recrystallization which can be controlled to yield suitable grain sizes. Within a suitable sequence of thermomechanical treatments, the hot-worked structure can be retained to give improved mechanical properties including creep resistance.

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