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Structural and substructural observations during thermomechanical processing of two ferritic stainless steels

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The substructural and structural features of two ferritic stainless steels were investigated and their variation with process variables noted. The influence of certain structural features on recrystallization and grain growth are discussed.

1. Introduction
Since the early part of this century the use of stainless steel has been extensive. The addition of nickel as an alloying element to steel improved the mechanical and corrosion properties to the extent that the austenitic grades achieved marked domination. A serious world shortage of nickel prompted more research into the ferrite grades [1], the major breakthrough occurring when Binder and Spendelow [2] discovered that if the carbon and nitrogen contents could be maintained sufficiently low then properties were greatly enhanced. More economic production of these low interstitial grades have since increased their use considerably. Typically, the carbon + nitrogen content must not be greater than 0.1%. The more common ferritic steel grades type 430 contain 17% Cr and recently this has been supplemented by type 18-2 containing 18% Cr and 2% Mo which it is claimed improves both mechanical and corrosive properties. Thus the major advantage of the ferritic steels is their relatively low cost but their stability and the lack of transformation products render them particularly susceptible to grain growth at elevated temperatures. There is little information in the literature concerning the hot working of the ferrite grades particularly in relation to their structural and substructural behaviour. This communication reports on the structural and substructural observations during and subsequent to the hot rolling of type 430 and type 18-2 ferritic steel.

2. Experimental details
Materials was supplied from BSC Swinden Laboratories in the form of 65 mm × 25 mm hot-rolled stock. Type 430 and type 18-2 steels were available and their chemical composition is given in Table I. The as-delivered stock was machined to 20 ± 0.5 mm after sectioning into 180 mm lengths. These bars were then given a leading edge taper of 20° extending about 25 mm along the length to facilitate entry to the roll gap.

The range of hot-rolling temperatures investigated was 800 to 1100° C in 25° C intervals. A strain rate of about 6 sec^{-1} was maintained and determination of the structure prior to entry in the roll gap was obtained by a series of annealing tests from which it was established that a soak of 20 min prior to deformation gave a fully recrystallized structure.

In order to reduce deformation gradients over the thickness of the slab during rolling, reductions were kept as large as possible. Hence the rolling schedule consisted of a 40% reduction followed by 20% which reduced the thickness to about 8 mm in the temperature range 925 to 1100° C. A single pass 50% reduction was imparted to material in the range 800 to 925° C using an original thickness of 10 mm. The interpass time was less than 30 sec and after the second pass the material was rapidly quenched to room temperature to retain the deformational structure.

3. Results and discussion
3.1. Hot rolling
At rolling temperatures of about 1100° C optical micrographs of ferritic stainless steels revealed elongated pancake grains containing a well-defined substructure within the original boundary walls. Fig. 1 is an optical micrograph of type 18-2 steel whilst Fig. 2 illustrates the internal structure of type 430 which is often referred to [3–5] as veining and is due to the

TABLE I Chemical composition of alloys

Alloy	Cr	Mo	C	N	Si	Mn	Ni	S	P
Type 430	16.7	–	0.008	0.026	0.24	0.62	–	0.006	0.012
Type 18-2	18.0	1.99	0.006	0.025	0.30	0.55	–	0.007	0.011

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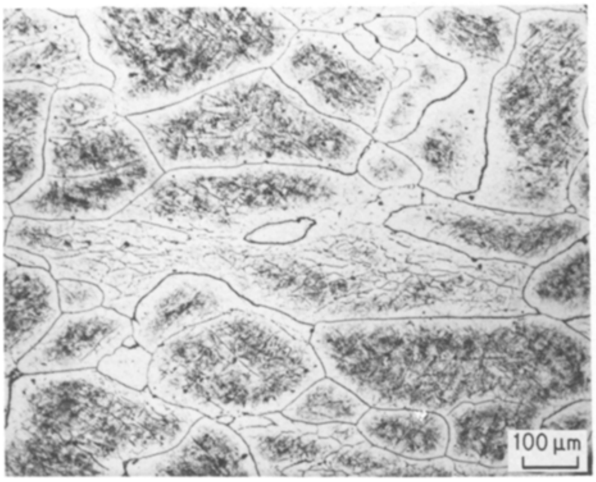


Figure 1 Structure of annealed type 18-2 steel.

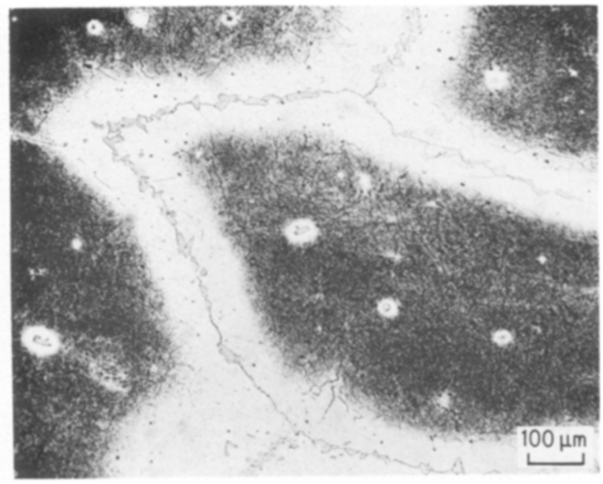


Figure 3 Microstructure of type 430 steel hot rolled at 1100° C.

production of highly stable subgrains. Such veining occurs irrespective of the propensity to form austenite at deformation temperatures which would, of course, be visible as martensite after cooling. Most of the martensite observed was sited at grain boundaries although localized distributions could be detected at other structural heterogeneities such as inclusions (an example of this feature is shown in Fig. 3). The extent of austenite present depends upon the chemical composition and whether the process route passes through the austenite loop (Fig. 4) [6]. Various empirical formulae exist [7, 8] to predict the level of austenite present during cooling at a specific temperature. The formulae are based upon the contributions of the ferritic forming elements (chromium, molybdenum, silicon) and the austenite stabilizing elements (carbon, nitrogen, manganese). Martensite is readily distinguishable from the ferritic phase in the TEM and appears as laths containing a relatively high dislocation density (Fig. 5). SADPs from these areas revealed a streaked b c c pattern indicating distortion of the b c c lattice. The regression of the austenite/martensite formed ferrite and $M_{23}C_6$ carbides.

It is well established [9] that during hot working the formation of subgrains within the original grain structure occurs because of the generation of dislocations which is illustrated in Fig. 6 for Type 430 steel. Their generation is directly related to the applied stress and

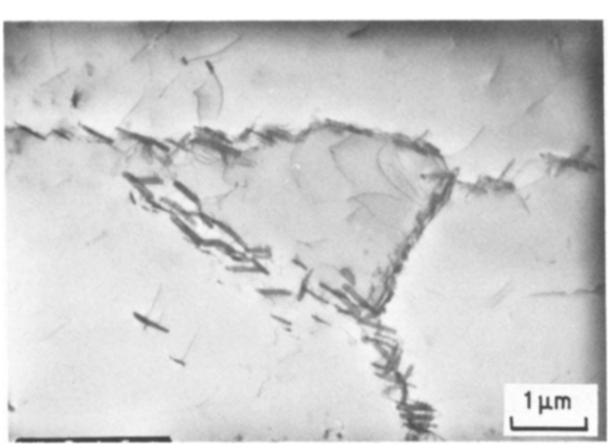


Figure 2 Veining in type 430 steel.

their subsequent rearrangement by glide, climb and cross-slip results in the typical three-dimensional subgrain arrays. The development of this substructure during rolling has been reported previously [10] and it has been shown that such dynamic recovery is the only softening mechanism operating during the deformation of ferritic steels.

Fig. 3 indicates that at higher rolling temperatures the structure in the region of grain boundaries may be heterogeneous. The austenite or carbides which form at the boundaries deplete the surrounding areas of carbon/chromium and create relatively precipitate-free zones. This may lead to poor corrosion and mechanical properties [4] but the zones also encourage rapid recrystallization after deformation until the carbide distribution becomes effective in pinning the moving grain boundary as shown in Fig. 7. Higher rolling temperatures also result in fine precipitation occurring on dislocation networks (Fig. 8); the networks varying in size and indicating the relative stability of the boundary (Fig. 9). The narrower the width of the network and the smaller the distance between dislocations indicates a greater proportion of

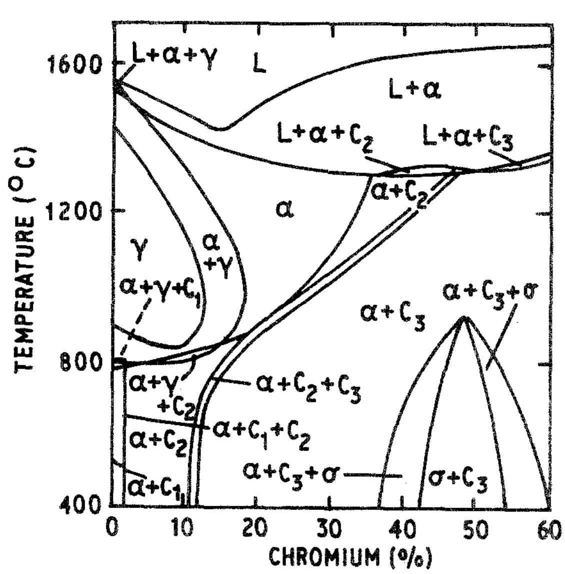


Figure 4 Fe–Cr–C equilibrium diagram at 0.1% C. $C_1 = M_3C$, $C_2 = M_7C_3$, $C_3 = M_{23}C_6$.

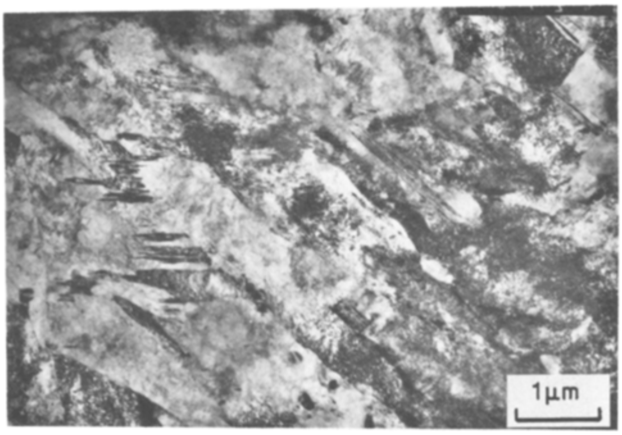


Figure 5 Martensite in type 430 steel.

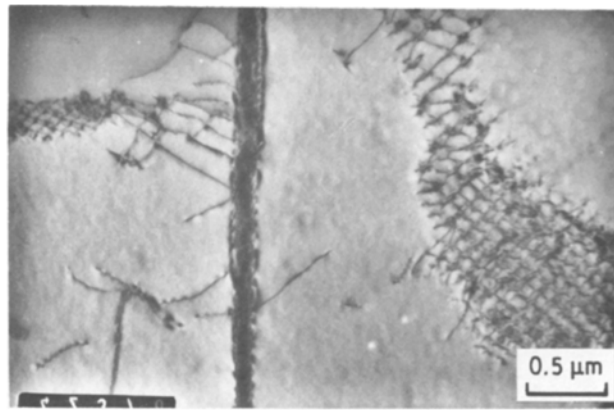


Figure 8 Substructure of type 18-2 hot rolled at 1100° C and quenched.

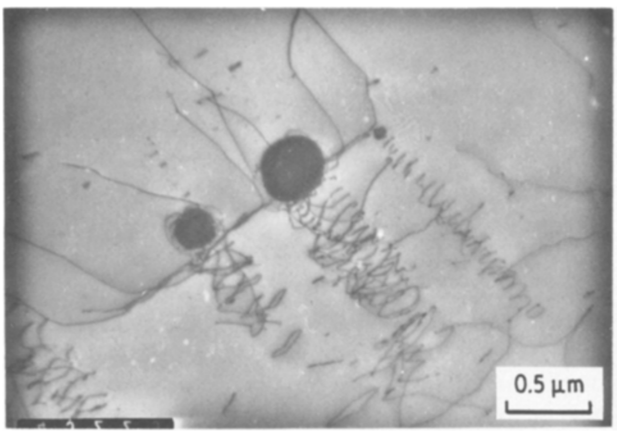


Figure 6 Substructure of type 430 steel hot rolled at 850° C and annealed 20 min at 825° C.

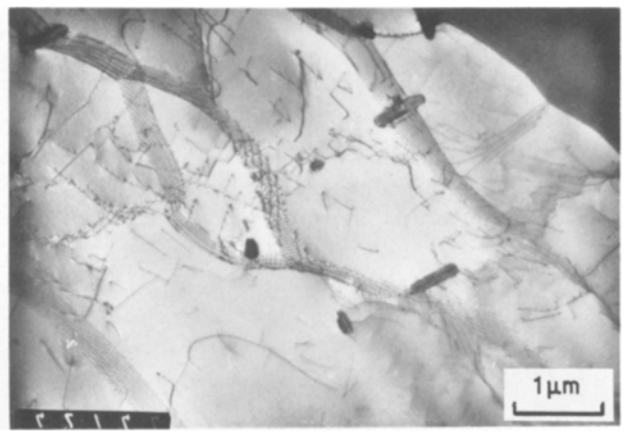


Figure 9 Dislocation networks in type 18-2 steel.

substructural inhomogeneity and the chance of formation of a subgrain is increased. A typical subgrain structure observed in both steels is shown in Fig. 10.

Substructural features are related to the hot-working parameters, temperature and strain rate. Hence if temperature gradients exist through the thickness of the rolled specimens then so will substructural variations. It was generally observed that recrystallization after deformation produced larger grains at the centre than at the surface which were clearly connected with substructural inhomogeneities including deformation bands (shown for type 18-2 in Fig. 11a and 430 in Fig. 11b) which were only observed close to the surface of the specimens.

As the deformation temperature was decreased to below 900° C the amount of precipitates observed in the structures increased significantly (Fig. 12). At these temperatures retained austenite commences to regress to $M_{23}C_6$ and ferrite. These sites act as stress concentrations and deformation is more concentrated at these areas of structural heterogeneity.

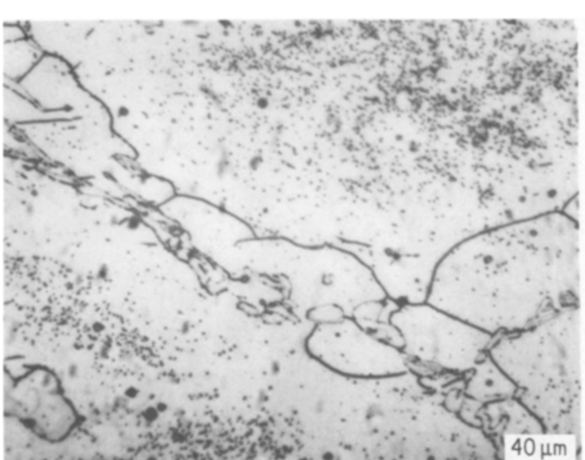


Figure 7 Microstructure of type 430 hot rolled at 1000° C.

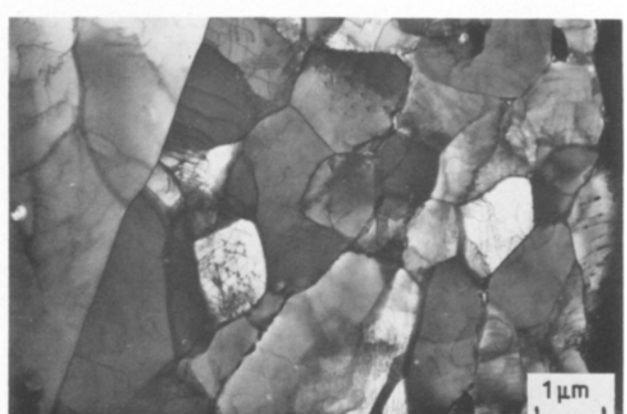


Figure 10 Substructure of type 18-2 hot rolled at 1050° C and quenched.

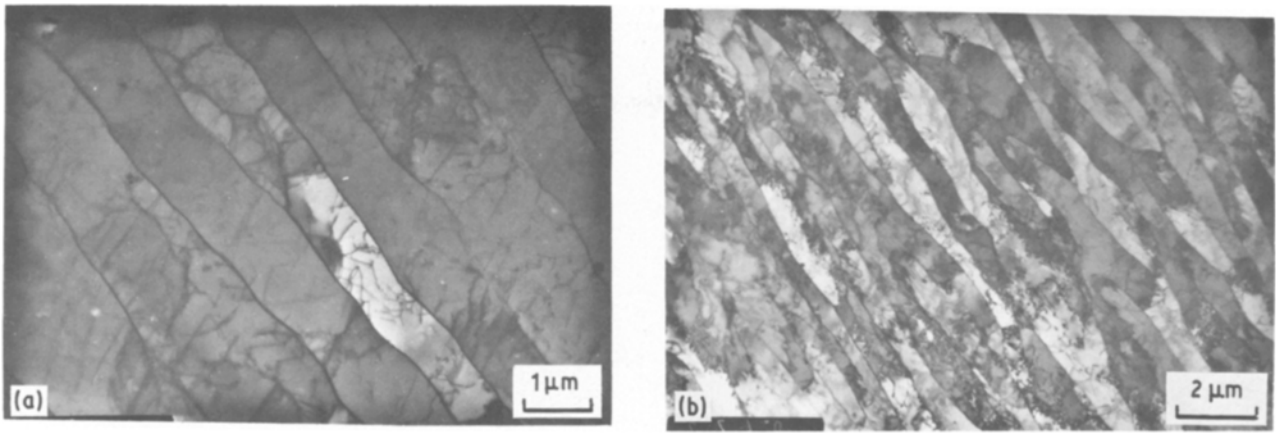


Figure 11 (a) Substructure of type 18-2 hot rolled at 1025° C and quenched. (b) Substructure of type 430 hot rolled at 1025° C and quenched.

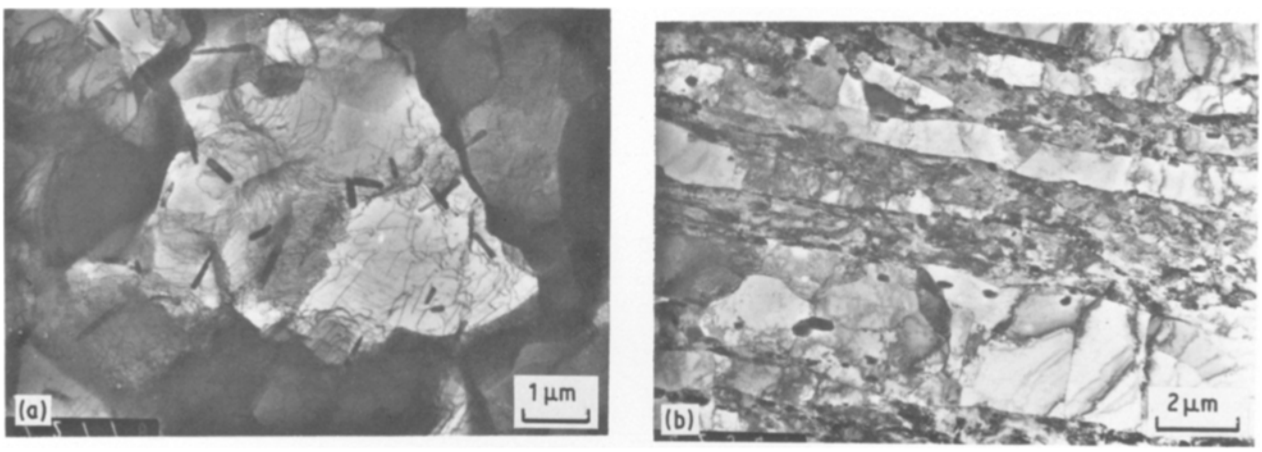


Figure 12 (a) Substructure of type 18-2 hot rolled at 850° C and quenched. (b) Substructure of type 430 hot rolled at 900° C and quenched.

Figure 13 Grain-boundary nucleation in type 430 steel.

Such areas will act as prime nucleation sites for the recrystallization process.

3.2. Recrystallization

Nucleation of recrystallization was observed to occur primarily at the deformed original grain boundaries, Fig. 13, and at triple point junctions (Fig. 14). The type 430 steel was observed to contain austenite,

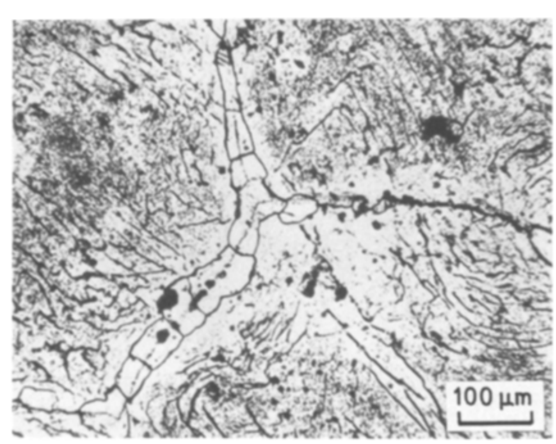
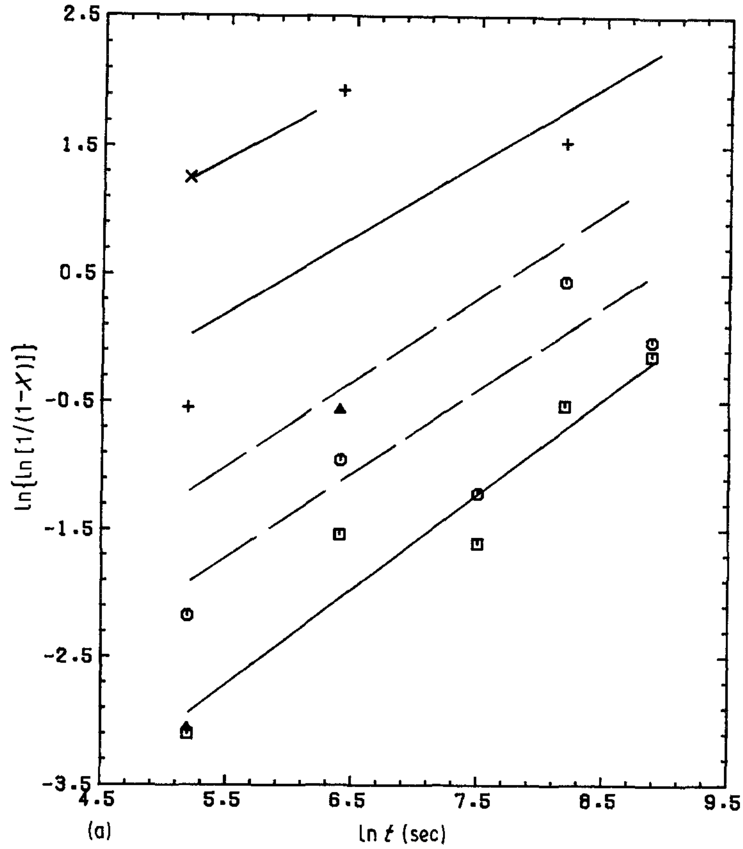


Figure 14 Grain-boundary nucleation in type 18-2 steel.



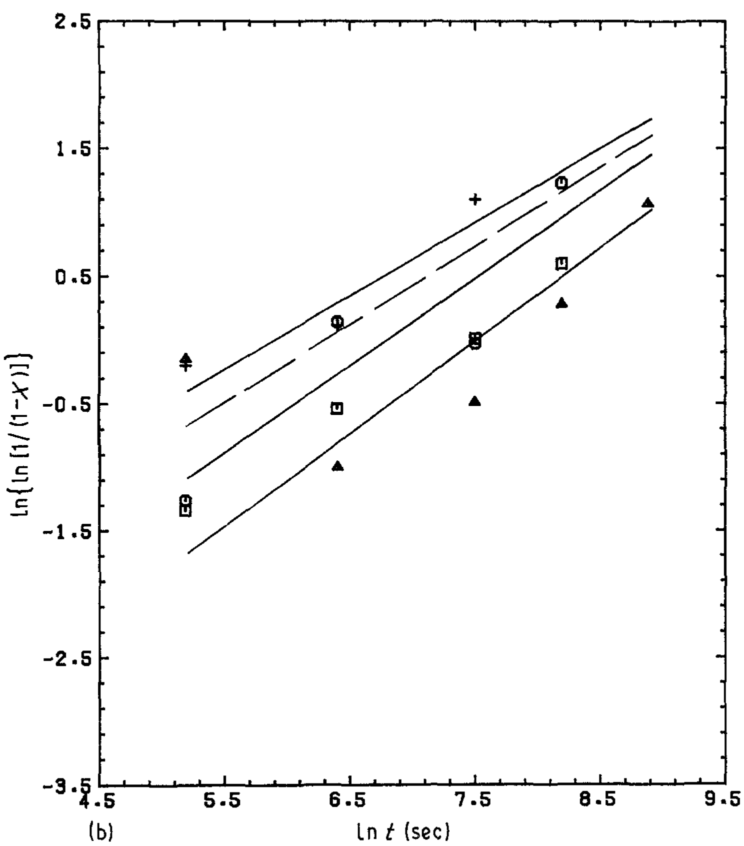


Figure 15 Recrystallization kinetics in (a) type 18-2 (□) 900° C, (○) 950° C, (△) 1000° C, (+) 1050° C, (×) 1100° C; (b) type 430 (□) 900° C, (○) 950° C, (△) 1000° C, (+) 1050° C.

decorating grain boundaries, which would also promote recrystallization at these sites because austenite, being harder than ferrite at elevated temperatures, will tend to concentrate deformation at its locale. The chance of forming nuclei at these sites is thus increased. There is therefore the implication that greater amounts of austenite promote a greater number of recrystallization nuclei and hence the probability of a more random texture. This would benefit forming and more importantly roping behaviour [11]. Similarly, a smaller grain size prior to rolling thus increasing the deformed grain area might aid finishing operations. Gladman *et al.* [12] has observed that in ferritic steels second phase particles of 0.5 to 20 μm diameter may accelerate recrystallization by acting as nucleation sites but particles smaller than 0.2 μm have little effect. In general large particles were not observed in the steels under investigation in this work although

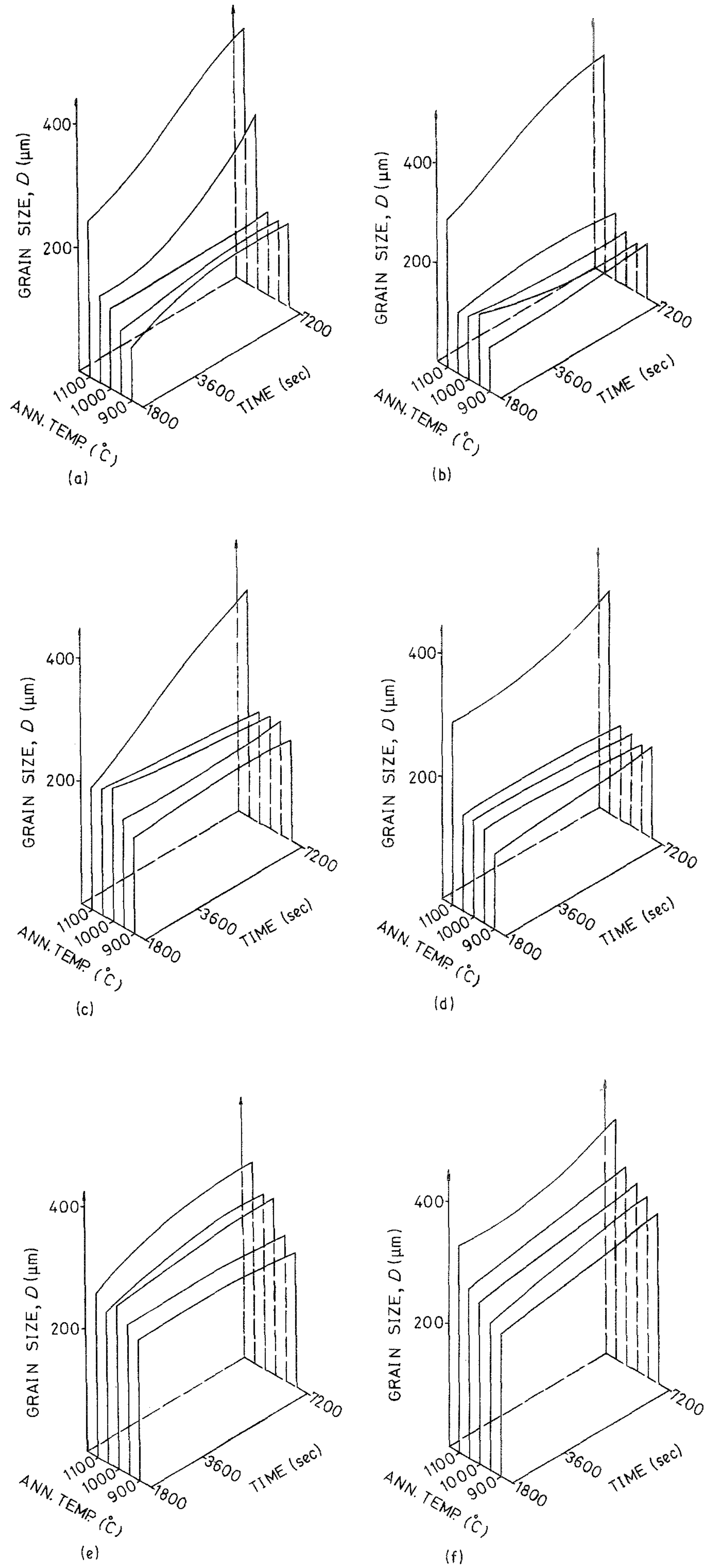
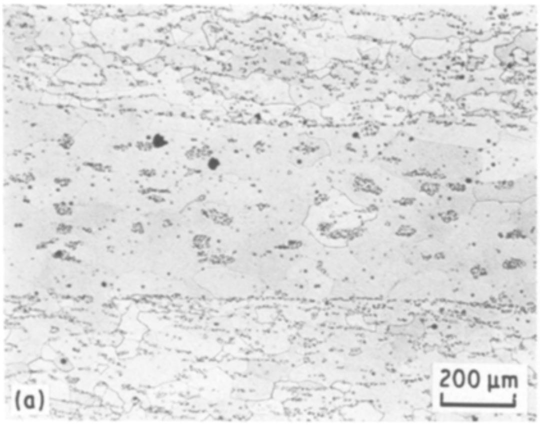


Figure 16 Effect of subgrain size on recrystallised grain size. (a) type 18-2, hot rolled 925° C, subgrain size 0.838 μm. (b) type 430, hot rolled 925° C, subgrain size 1.283 μm. (c) type 18-2, hot rolled 1000° C, subgrain size 1.326 μm. (d) type 430, hot rolled 1100° C, subgrain size 1.364 μm. (e) type 18-2, hot rolled 1100° C, subgrain size 2.240 μm. (f) type 430, hot rolled 1100° C, subgrain size 1.910 μm.



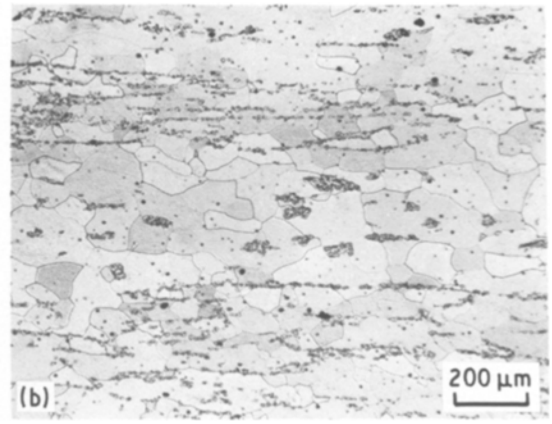


Figure 17 (a) Microstructure of type 430 hot rolled at 975° C and annealed 30 min at 1000° C. (b) Microstructure of type 430 hot rolled at 925° C and annealed 30 min at 1000° C.

several particles of about 0.2 μm could be observed as shown for example in Fig. 6.

Provided that the deformation conditions are suitable then static recrystallization under isothermal conditions can be described by the Avrami nucleation and growth equation:

$$X_v = 1 - \exp(-At^m)$$

where X_v is the volume fraction recrystallized in time t sec and m and A are constants. The data presented in Fig. 15 indicate that the time exponent m is approximately 0.65 for both of the alloys. For materials which dynamically recover [13] the expected value of m is about 2. Previous workers [13] have shown that the exponent is sensitive to grain size; larger grains lowering the value obtained for m. As nucleation and growth proceeds in areas of relatively high strain such as grain boundaries then strain gradients, across say a large grain, must alter the recrystallization kinetics. The grains presented to the hot mill in this work were, in fact, abnormally large ($\approx 200\,\mu$m) and these large grains typical of a hot-worked ferritic structure and frequently of the cube-on-face texture [15] have the lowest stored energy and hence recrystallize slowly. This would certainly contribute to a lowering of the time exponent m.

The hot-work-induced subgrain size also affects the recrystallized grain size. Some of the hot-rolled material exhibiting differing subgrain sizes was subjected to similar annealing treatments (Fig. 16). The recrystallized grain size D varied with the subgrain size d as well as the time t and temperature T. This would suggest that certainly not all of the nucleation sites are at original (elongated) grain boundaries nor at second-phase particles. Sheppard and Zaidi [16] have related the recrystallized grain size D with the subgrain size d in aluminium alloys which also deform by dynamic recovery and associate this with nucleation by subgrain coalescence. The time per subgrain coalescence will decrease with the decrease in area of a subboundary wall which is in contact with its neighbour. Thus if we assume that a particular number of coalescences are required to form a high-angle boundary and hence a nucleus capable of migrating then the rate of nucleation will increases with decreasing subgrain size due to the decrease in contact area per subgrain. Figs 17a and b illustrate the differences caused by varying hot-rolling parameters whilst Fig. 18 shows that large cube-on-face pancake grains are usually the last to recrystallize.

When recrystallization has been nucleated the growth of the newly formed grains may be impeded by obstacles such as other moving grain boundaries, second-phase distribution, etc. The type 430 steel was observed to be particularly susceptible to the second-phase distribution. Fig. 19 shows the effect of annealing temperature on hot-rolled stock subjected to identical processing conditions. The recrystallized grain size remains relatively stable up to a temperature of about 850° C. Below this temperature the structure consists essentially of ferrite plus carbides in which the carbides act to pin moving grain boundaries as shown for example in Fig. 20. When the temperature is increased austenite commences to form from the dissolution of the carbides and the pinning effect is reduced. These areas of carbides also lead to inhomogeneous subgrain sizes during deformation (Fig. 21) and hence heterogenous recrystallized grain structures.

We conclude that for various reasons the recrystallized grain size in ferritic steels may be extremely heterogeneous and although maximizing the total strain may partially alleviate this problem, maximum grain refinement cannot be achieved in this way. It will

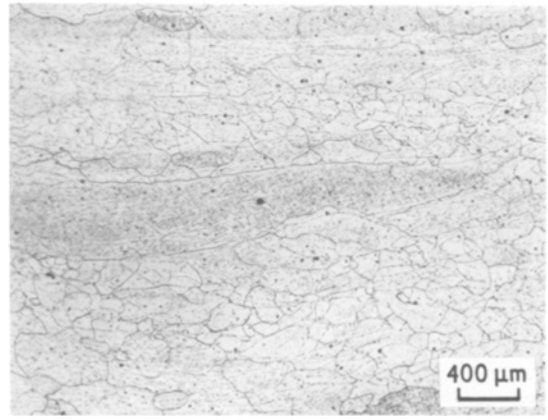


Figure 18 Microstructure of type 18-2 hot rolled at 925° C and annealed 30 min at 900° C.

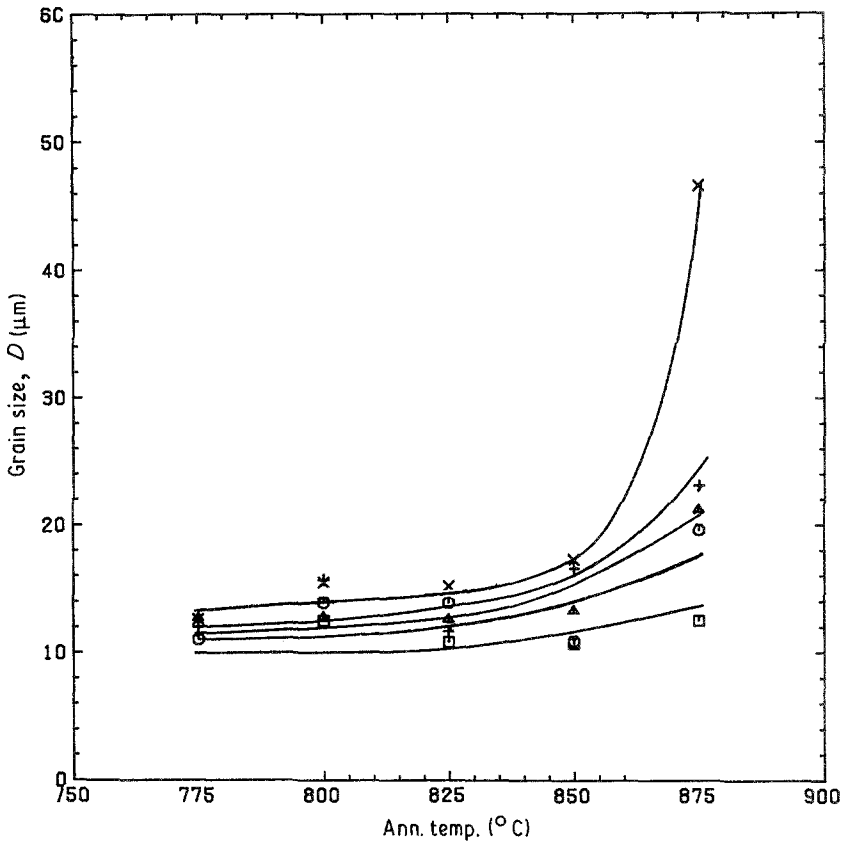


Figure 19 Variation in grain size prior to the hot rolling process. Type 430, (□) 180S (○) 600S (△) 1200S (+) 2400S (×) 5400S.

depend upon the conditions of deformation, the initial grain size, the extent of second phase distributions and the cooling rate.

3.3. Grain growth

During recrystallization the driving force for grain-boundary migration is supplied by the energy stored during the hot-working process; when the period of grain growth commences it is the reduction in energy which occurs with reduction in grain-boundary area.

Grain growth in ferritic steels is impeded by the pinning of moving grain boundaries by second-phase distributions. In many cases boundaries were observed to bow out between pinning points, illustrated for example in Fig. 22. Where austenite decorated the boundaries of the deformed grains, the new grains acquired the dimensions of the decorating bands. Rapid heating to high temperatures (> 1100° C) during an annealing cycle in order to solutionize the austenite produced a more homogeneous recrystallized grain distribution but of larger size (Fig. 16 for example). During growth any attempt by the grain boundary to escape the pinning particles results in a local increase in energy and a drag effect can clearly be observed on the migrating boundary (Fig. 22). The energy required to overcome such barriers may be greater than that for simple thermal activitation. This energy must be created during growth by the elimination of grain interfaces by the growing grain such that unpinning may occur. The only other mechanism under which grain growth could proceed would be a coarsening of particles since Gladman [17, 18] has shown that this would aid the growth mechanism.

Secondary recrystallization can also occur in ferritic

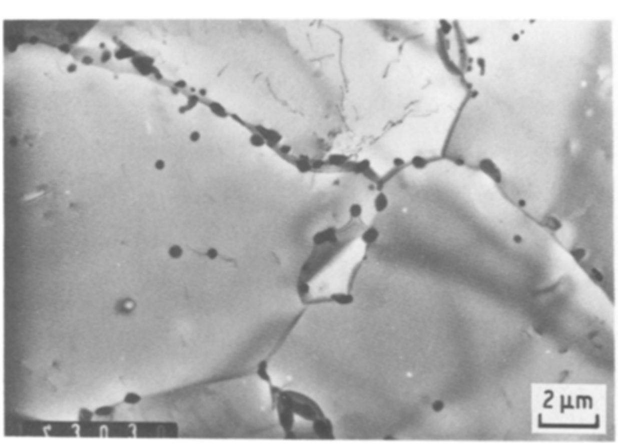


Figure 20 Grain-boundary properties in type 430 hot rolled at 1000° C and annealed for 20 min at 825° C.

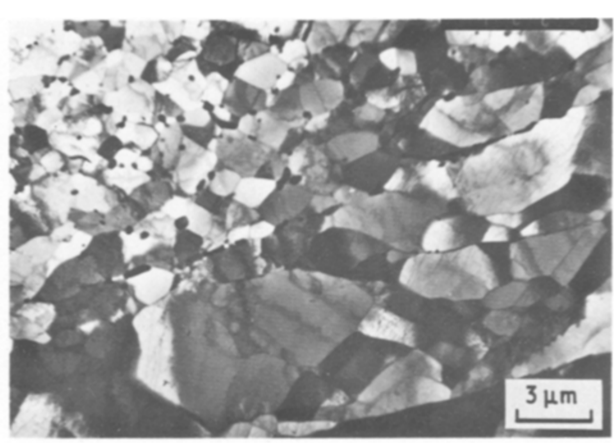


Figure 21 Banding in type 430 hot rolled at 825° C and held for 3 min at 825° C.

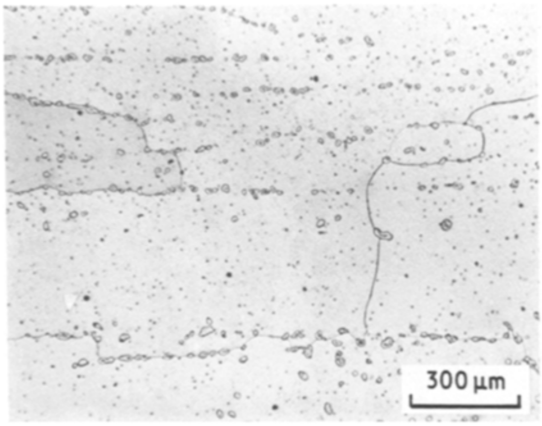


Figure 22 Type 18-2 hot rolled at 1000° C and annealed 60 min at 1100° C.

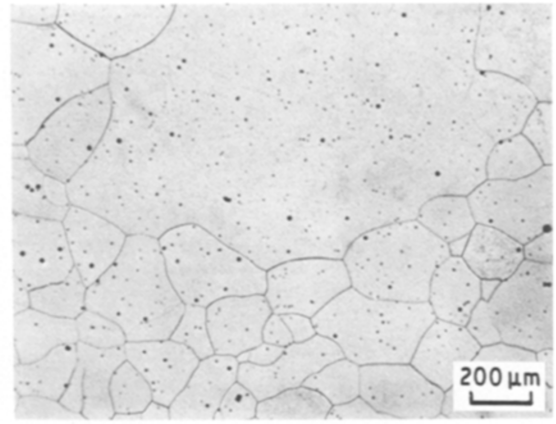


Figure 23 Type 430 hot rolled at 1050° C and annealed for 20 min at 1050° C.

stainless steels both at higher temperatures (Fig. 23) when most of the carbon is in solution and at lower temperatures where pinning carbides may coalesce. Grain coarsening is aided in these steels by the additions of elements which only dissolve at higher temperatures (e.g. niobium, titanium).

4. Conclusions

1. Ferritic stainless steel exhibits a veined structure following hot working.

2. Deformation during hot working is inhomogeneous, particularly at grain boundaries exhibiting austenite.

3. Below approximately 900° C austenite regresses to ferrite plus $M_{23}C_6$ carbides.

4. Recrystallization initially occurs primarily at previously deformed grain boundaries but must later be added by subgrain coalescence.

5. The Avrami time exponent, m, is reduced when recrystallizing large grain-sized stock.

6. The recrystallized grain size depends on time, temperature and subgrain size.

7. Second-phase particles serve to pin both moving recrystallized grain boundaries and grain boundaries during growth.

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