JOURNAL OF MATERIALS SCIENCE 8 (1973) 1413-1420

The reversion of martensite to austenite in certain stainless steels

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An investigation has been made of the reversion of martensite (α') to austenite (γ) in two stainless steels (i) Fe-16 wt % Cr-12 wt % Ni (of low interstitial content) (ii) Fe-15 wt % Cr-8½ wt % Ni-2 wt % Mo-0.09 wt % C. The alloys were refrigerated to produce ~ 12 to 15% martensite (α') and then heated for short times at various temperatures ranging from below A_s to above A_f. With rapid heating the reversion of α' to γ occurs largely by a shear mechanism. In the Fe-16Cr-12Ni alloy individual grains of α' transform to grains of reversed γ of similar size and shape. In the carbon-containing alloy there is evidence of break-up of the α' grains on reversion. An increase in the strength results from reversion and this is attributable mainly to the high dislocation density of the reversed γ.

1. Introduction

The reversion of martensite to austenite and the resultant strengthening that can be achieved have been studied by many investigators in a range of steels (e.g. [1-10]). It has been shown, for example, that the transformation commonly occurs by a diffusionless mode and that the "reversed" austenite contains a high density of defects. From the practical viewpoint it is of interest to consider the effectiveness of reversion treatments as a means of strengthening steels. The strengthening effect in the austenite depends in a complex manner on many factors, relating to alloy constitution and structure, and to heat treatment conditions, as follows.

1. The volume fraction of martensite present prior to reversion which determines the volume fraction of reversed austenite produced.
2. The type of martensite present (e.g. plate or lath; internally twinned or with a dislocation sub-structure); this may affect the density and distribution of defects in the reversed structure partly by the inheritance of defects, and partly by the nature of the dislocation movements occurring in the reverse transformation.
3. The structural condition of any austenite present prior to reversion, e.g. the presence of dislocations introduced into the austenite by the initial formation of martensite or by cold working.
4. The magnitude of the volume contraction accompanying the bcc/fcc change; this is relevant to the generation of vacancies and hence to the detailed nature of the resultant dislocation sub-structure.
5. The alloy constitution with respect to the occurrence of diffusional reactions, such as precipitation of carbides or intermetallic compounds. Precipitation in the martensite during heating for austenitizing or during a controlled pre-treatment may affect the reverse transformation in terms of dislocation movement and generation, and can also contribute to the strength of the reversed austenite. Also if the reversed austenite is supersaturated, e.g. with carbon, then precipitation may occur on the nucleation sites provided by the dislocations present. Other factors affecting the extent to which diffusional processes occur include the diffusion coefficients of the various elements, the heating rate, the temperature change over which the reverse transformation occurs (i.e. A_s-A_f), and the austenitizing temperature and time.
6. The recovery and recrystallization behaviour of the reversed austenite. This depends on numerous factors such as tempera-

ture, time, diffusion coefficients, stored energy, stacking fault energy, type of sub-structure, effects of solute atoms or precipitates.

7. The number of cycles of reversion treatment to which the alloy is subjected; the cumulative strengthening effect is influenced by factors such as the amounts of martensite formed on cooling after reversion (stabilization affects this) and the regions in which such martensite forms (i.e. the untransformed or reversed austenite regions).

Much of the published work has been concerned with alloys based on the iron-nickel system but information on reversion in stainless steels is also available [2, 11-17]. The results reported here form part of a detailed study to determine the reversion behaviour of two semi-austenitic stainless steels in which the martensite morphology and also the stacking fault energy of the γ differ from those of iron-nickel alloys.

One alloy (A) had a composition of Fe-16.1 wt % Cr-11.7% Ni, with a low interstitial content (~ 0.003 % C and 0.009 % N). The other alloy (B) contains molybdenum and carbon (Fe-14.9 wt % Cr-8.7 wt % Ni-2.05 wt % Mo-0.09 wt % C-0.01 % N) for the study of possible effects of carbon precipitation in relation to reversion. In both alloys the impurity levels were low, e.g. Si + Mn (~ 0.1 wt %), S + P (~ 0.01 wt %). The alloy compositions were chosen to give austenitic structures at room temperature, free from δ-ferrite, and to have M_S temperatures not far below room temperature.

2. Experimental procedure

The alloys were vacuum melted and cast by International Nickel Ltd and after forging into plate, they were homogenized for 24 h at 1100°C. Cold-rolling, with appropriate interstage softening heat-treatments, was used to produce sheet samples of various thicknesses, down to ~ 0.06 mm. Solution treatments were carried out with specimens sealed in silica tubes under a partial atmosphere of argon; for alloy A the treatment was 20 min at 1050°C and for alloy B, 1 h at 1250°C, in both cases followed by water-quenching. The austenite grain size was ~40 μm. After heat-treatment, surface layers were removed by chemical thinning. Samples were cooled to − 196°C and held for 15 min (alloy A) and 1 h (alloy B). Reversion treatments were carried out using salt bath heating.

Measurements of martensite content were made using a Sucksmith balance; the reproducibility of measurements on a standard iron specimen was of the order of 1 %, which corresponds to 0.1 % martensite in a steel with a total martensite content of 10%. Specimen to specimen variation in martensite content of the alloys studied was of the order of 2½ %, i.e. individual specimens could vary by ~ 0.4 % martensite at the 15 % martensite level.

Hardness measurements were used to complement the magnetic measurements in following the progress of reversion, and to indicate the strengthening effect; some tensile measurements were also made. Dilatometry, using a sensitive differential instrument, was also used to a limited extent to study the kinetics of reversion on continuous heating.

Structural features were examined by light and electron microscopy; for the latter, thin foils were prepared by a disc technique involving jet profiling using a solution of 37 % HCl. Final thinning for alloy A used a saturated solution of chromium trioxide in orthophosphoric acid, cooled by an ice/water mixture at 13 V; for alloy B the final thinning used a solution containing 133 cc glacial acetic acid, 25 g chromic oxide and 7 cc of distilled water at 10 to 15°C and 22 V.

3. Results

3.1. The direct martensite transformation

The M_S temperatures after the standard solution treatment were determined as −50 and −40°C for alloys A and B respectively. Refrigeration of alloy A at − 196°C for 15 min produced ~ 15 % ferromagnetic martensite, α'. The refrigeration treatment of 1 h at −196°C used for alloy B, produced 12.5 % α'; (in this alloy, holding for 8 h at −196°C produced 15 % α'). The increase in hardness on refrigeration was ~ 75 HV in alloy A and ~ 40 HV in alloy B, so that the presence of carbon does not appear to have a substantial effect on the hardness of the α'.

The α' forms as lath-like grains containing a high dislocation density [18-22]. Groups of these grains form in "sheets" bounded by $\{111\}_\gamma$ planes (e.g. Fig. 1) and regions of ϵ martensite and/or stacking fault clusters are also present within these sheets. The retained γ has a low dislocation density.

3.2. The effect of reversion treatments on hardness and martensite content

Samples with partially martensitic structures produced by refrigeration were heated for short times (2 min for alloy A and 1 min for B) at various temperatures, the shorter time used for

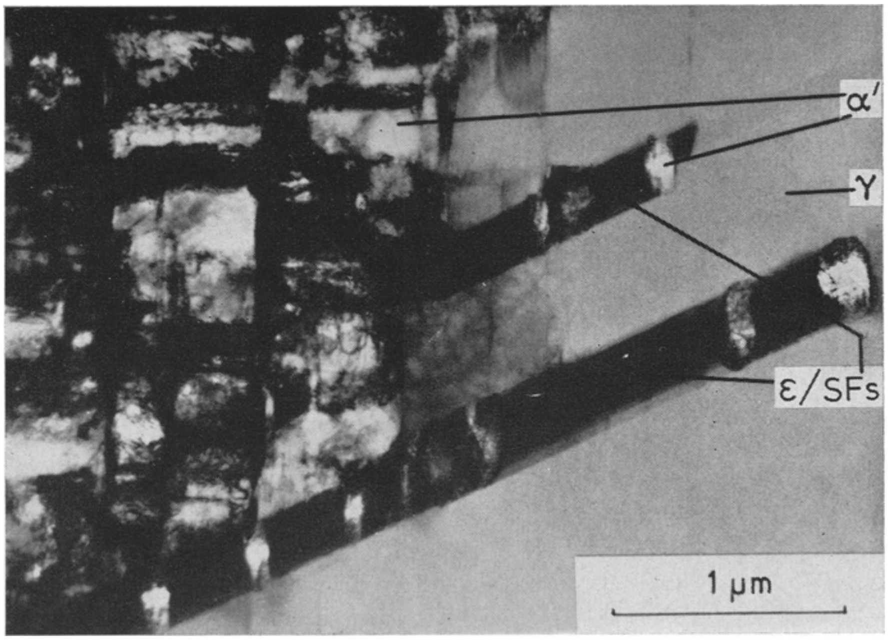


Figure 1 Alloy A Fe-16 wt % Cr-12 wt % Ni. Partially martensitic structure produced by cooling to −196° C.

alloy B aiming to minimize the possibility of carbide precipitation.

In alloy A, after a slight hardness increase in the range 450 to 500° C, the hardness fell to a plateau between 600 and 700° C before further decreasing towards the hardness of the original austenite (Fig. 2). After reversion at 600 to 700° C the austenite hardness was ~ 155 HV as compared with 100 HV for the original annealed austenite while the 0.2% proof stress (~ 220 MN m^{-2}) was almost double the original value (~ 115 MN m^{-2}). There was a small increase in α' content after heating between 300 and 400° C (Fig. 2) but the amount of α' decreased markedly above 500° C, and the A_s may be taken as 500 to 520° C; the A_f is estimated as 600° C.

The reversion characteristics of alloy B (Fig. 3) with respect to changes in hardness and α' content showed similarities to those of alloy A; however, the A_s and A_f temperatures were higher, ~ 570 and 775° C respectively, and the hardness decrease occurred over a wider range than in alloy A.

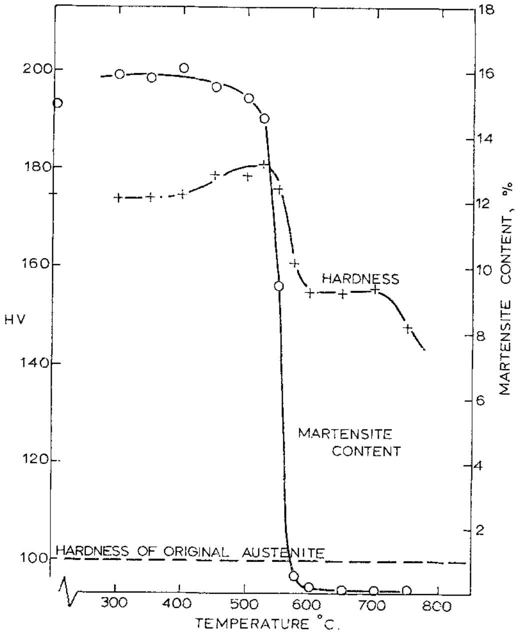


Figure 2 Alloy A Fe-16Cr-12Ni changes in hardness and martensite (α') content after heating the partially martensitic structure for 2 min to the temperatures shown and water quenching to room temperature.

3.3. Dilatometric results for reversion

Dilatometric experiments were made, using

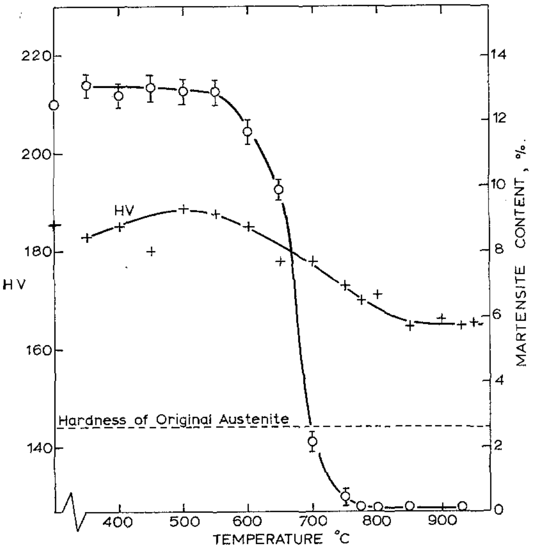


Figure 3 Alloy B Fe-15 wt % Cr-8½ wt % Ni-2 wt % Mo-0.09 wt % C. Changes in hardness and martensite (α') content after heating the partially martensitic structure for 1 min to the temperatures shown and water quenching to room temperature.

continuous heating of refrigerated samples at rates of 4.5, 16 and 165°C min^{-1} respectively. With the fastest of these rates the A_S temperatures were measured as ~ 540 and 550°C for

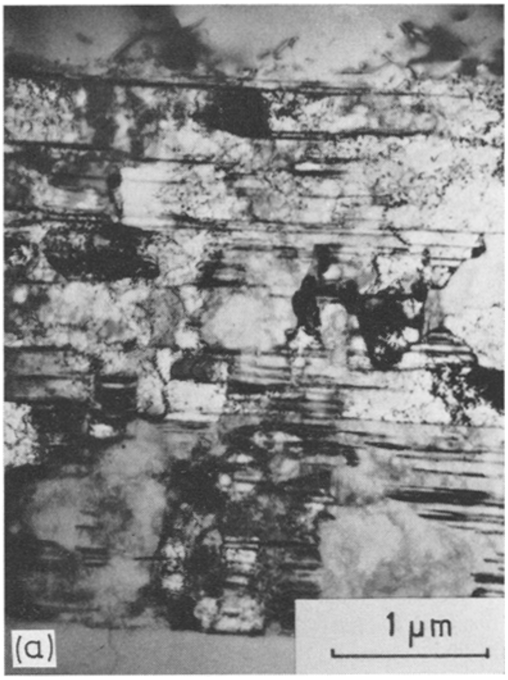
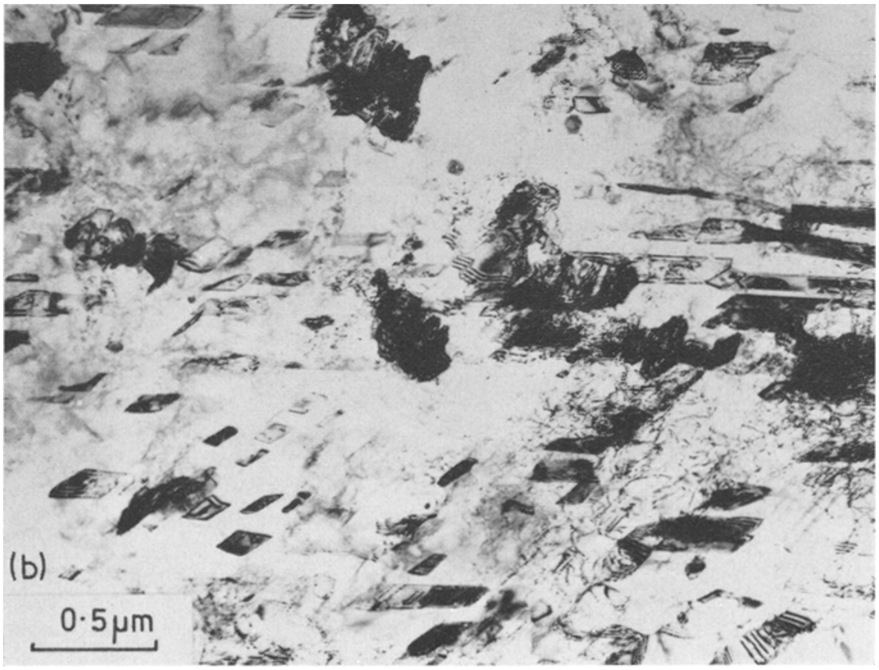


Figure 4 (a) Alloy A Fe-16 wt % Cr-12 wt % Ni. Structure after heating partially martensitic specimen for 2 min at 600° C showing regions of reversed γ bounded by retained γ. (b) Alloy B Fe-15 wt % Cr-8½ wt % Ni-2 wt % Mo-0·09 wt % C. Structure of reversed γ regions produced by heating partially martensitic specimen for 1 min at 775° C



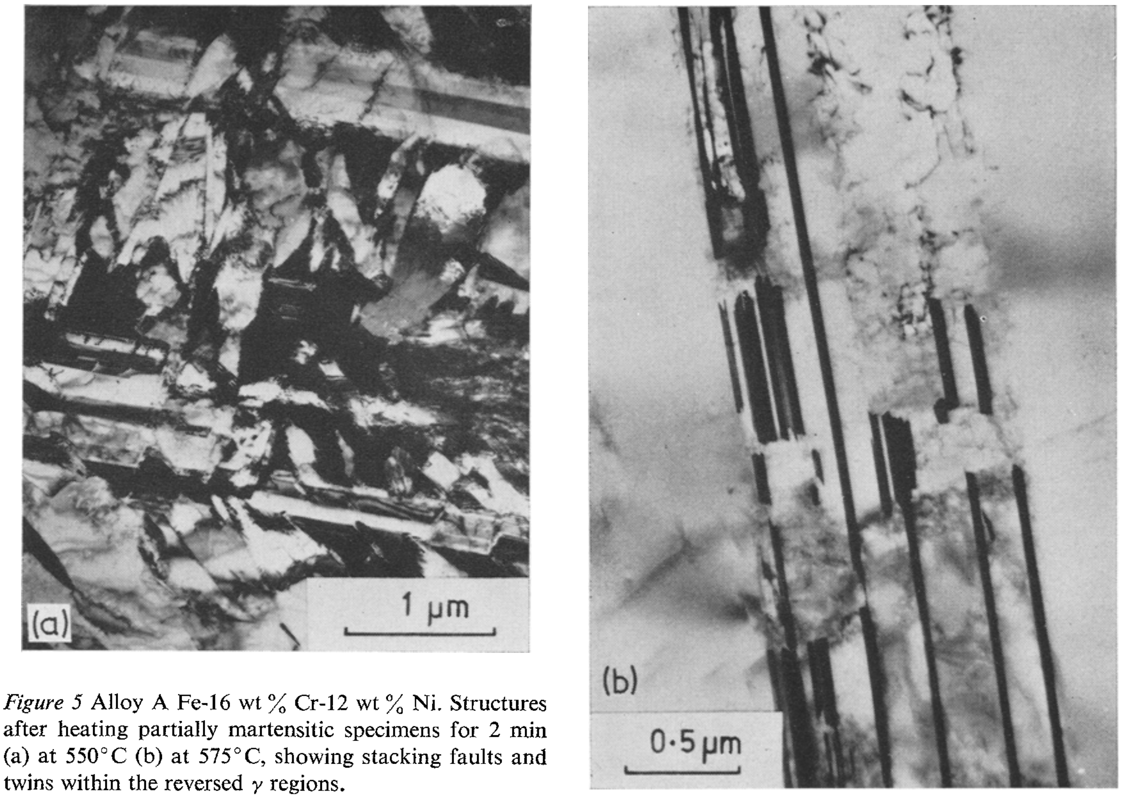


Figure 5 Alloy A Fe-16 wt % Cr-12 wt % Ni. Structures after heating partially martensitic specimens for 2 min (a) at 550°C (b) at 575°C, showing stacking faults and twins within the reversed γ regions.

alloys A and B respectively; with the slower rates the A_s was less sharply defined by the length changes, and the transformation appeared to begin at a lower temperature. The A_f temperatures were virtually independent of heating rate and were ~ 600 and 760°C for alloys A and B respectively.

3.4. Structural features of reversion

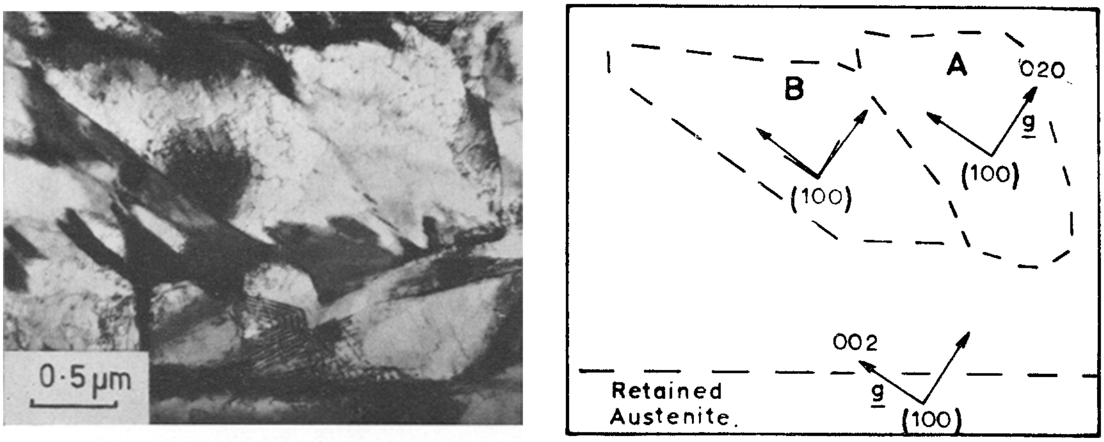
In both alloys, after reversion, light microscopy revealed "ghost" areas of "reversed" austenite which etched preferentially, and which corresponded to the original regions of martensite. These areas contained a high density of tangled dislocations, and numerous small twins and stacking faults (Fig. 4a and b); the faults were smaller and less frequent in alloy B than in alloy A. After heating alloy A for 2 min at temperatures between 550°C and A_f many stacking faults and twins were observed within the reversed γ areas (Fig. 5a and b).

In alloy A, examples were observed of areas in which the transformation produced reversed γ having essentially the same crystallographic orientation as the retained γ, e.g. Fig. 6. In alloy B there was evidence of a tendency for individual α' grains to produce, on reversion, several regions misoriented with respect to one another, e.g. Fig. 7; no evidence of carbide precipitation was obtained in samples heated for 1 min at the various temperatures studied.

4. Discussion

4.1. Mechanisms and kinetics of reversion

Evidence for the shear (diffusionless) nature of the reverse transformation is provided by the observation on alloy A that the reversed γ regions regain the orientation of the original (retained) γ (Fig. 6). This is in agreement with the findings of Breedis [15] and suggests that the same variants of the shears operate in the direct and reverse transformations. In the case of alloy A it appears that one grain of α' can retransform completely to a single "grain" of reversed γ, and it is possible that all the grains of α' within a sheet retransform at the same time; this would assist in the accommodation of the transformation shears. In alloy B the tendency for the break-up of individual α' grains during reversion may result from some carbon redistribution



(a)

(b)

Figure 6 Alloy A Fe-16 wt % Cr-12 wt % Ni (a) Structure after heating partially martensitic specimens for 2 min at 555°C, showing regions of reversed γ bounded by retained γ. (b) Data from diffraction pattern showing two reversed γ grains A and B having almost the same orientation as the retained γ; each region has a [100] zone axis and there is no measurable misorientation between the [001] directions in grain A and the retained γ, although a different reflection operates. The [001] direction in grain B is ~4° misoriented from the same direction in grain A and in the retained γ.

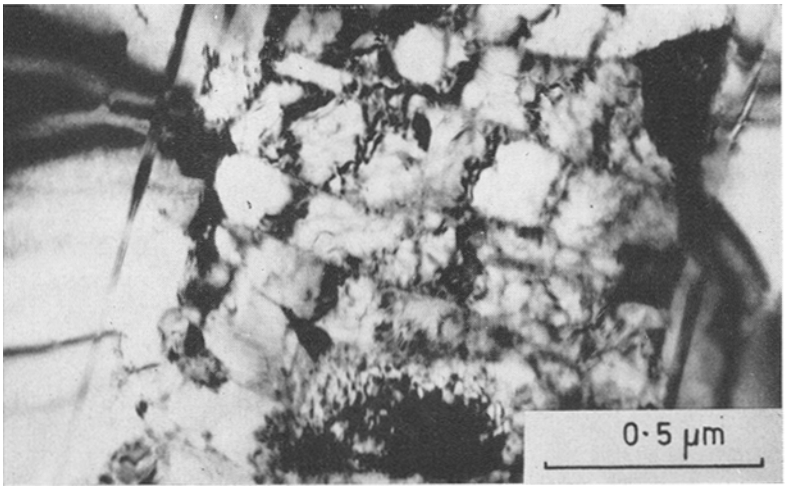


Figure 7 Alloy B Fe-15 wt % Cr-$8\frac{1}{2}$ wt % Ni-2 wt % Mo-0.09 wt % C. Structure after heating partially martensitic specimens for 1 min at 600°C (i.e. ~30°C above A_S). The α' grains in the area shown appear to have broken up, while other areas in the specimen appeared to be unaffected by the heating.

interfering with the shear process, and preventing entire grains of α' from undergoing the reverse shears of the direct transformation.

A point of interest concerning the A_S temperature in alloy A is that the magnetic data for specimens heated in a salt bath (Fig. 2) show some decrease in α' content below 500°C, whereas the dilatometry data indicate an A_S of ~540°C for the fastest heating conditions. However, specimens heated in a salt bath between ~300 and 450°C show a slightly greater α' content than that of the initial refrigerated state; a similar feature has been observed by Manganon and Thomas [27] on heating specimens of 304 stainless steel containing strain-induced α', and has been interpreted as a thermal nucleation effect associated with stress relief. It seems justified therefore not to take the A_S as

450°C [17] which is the temperature when the curve of α' content versus temperature first shows a downward trend (Fig. 2), but to take A_s as 500 to 520°C when the α' content begins to decrease below that of the original, refrigerated state; this value of A_s agrees reasonably with that measured by Coleman namely ~ 500 to 530°C.

In the dilatometric work the observed decrease in A_s with decrease in heating rates may result from the occurrence of a thermally activated mode of transformation [23-26]. In alloy B, the possibility of carbide precipitation during slow heating complicates the interpretation of the dilatometric results.

4.2. Fine structure of reversed austenite

The characteristic features of the "fine" structure of the reversed γ are a high density of tangled dislocations, together with small twins, and stacking faults, although the latter have largely disappeared once the reverse transformation is completed. The high dislocation density is likely to be partly inherited from the sub-structure of the α' crystals; however, a contribution to the dislocation density is expected from the various deformations which are associated with the reverse transformation and which take place in an already imperfect structure.

It is not clear from the observations whether the stacking faults (e.g. Fig. 5) participate in the mechanism of the reverse transformation or are formed by recovery processes subsequent to reversion. The smaller size of the faults and less frequent occurrence in alloy B than in alloy A, may be due to a shorter distance over which the leading partial dislocations can move before meeting an obstruction; this suggestion is consistent with the apparent greater fragmentation of reversed γ in alloy B.

The small twins which have been observed in the reversed austenite in both alloys, and by Breedis are considered to be an early stage in the recovery of the reversed austenite. A twin fault has roughly one half of the energy of a stacking fault because only half as many next-nearest neighbour bond violations are involved; thus the formation of a twin from stacking faults reduces the stored energy of the reversed austenite substructure. The relatively low stacking fault energy of the alloys favours stacking fault formation, and twins may form from stacking faults on adjacent planes. The contrast effects produced by the twins in electron microscopy suggest that they are very thin, and the considerable length (Fig. 5b) may be attributable to the very small differences in orientation between adjacent reversed austenite regions. The smaller size of the twins in alloy B is probably connected with the greater deformation of the reversed γ regions in this alloy.

4.3. Strengthening of austenite by reversion

The increase in hardness of the austenite achieved by reversion treatments just above A_f (namely from ~ 100 to 155 HV in alloy A and from ~ 145 to 170 HV in alloy B) is attributable to the defects of the reversed γ lattice; probably the main effect is the high dislocation density but the stacking faults and twins may also contribute significantly. There is no evidence that carbide precipitation is a contributory factor to strength; however, the slight hardness increase (Figs. 2 and 3) observed on heating refrigerated samples to ~ 500°C (i.e. before reversion occurs to any appreciable extent) may be associated with the segregation of carbon and/or nitrogen atoms to dislocations in the martensite.

The hardness levels after reversion are lower than those of the original refrigerated structures containing α'; this is partly attributable to the fact that the regions of reversed γ have the same crystal structure as the retained γ, and differ only slightly in orientation from one another and from the retained γ, so that less resistance to dislocation movement is offered as compared with the situation in the $\gamma + \alpha'$ structures.

The total strengthening effect of reversion in the present alloys is relatively small compared to that previously reported for alloys such as Fe-30% Ni: however, only 15% α' undergoes reversion compared with typical amounts of α' in excess of 90% for Fe-Ni alloys previously studied, and in fact, the specific strengthening effect (i.e. per % α') appears to be greater in the case of the stainless steels. Thus, in alloy A an increase in proof strength of ~ 100 MN m^{-2} results from the reversion of 15% α' whereas in an Fe-26% Ni-0.4% C alloy [28] the reversion of a similar amount of α' increases the proof stress by ~ 70 MN m^{-2}. In interpreting this effect the difference in α' morphology between these alloys should be borne in mind. The α' in the stainless steels forms in "sheets", whose total volume is considerably greater than the actual volume of α'. Thus the "effective" volume of the "ghost areas" after reversion treatments corresponds to the volume of the sheets rather than to the volume of α'; hence, for a given initial percentage of α' more effective hindrance to dislocation movements may be provided in the stainless steels after reversion as compared with the iron-nickel alloys. Further work, however, is required to clarify the influence of various structural features contributing to reversion strengthening in different types of alloys.

5. Conclusions

1. The reversion of α' to γ in the two steels studied, using salt bath heating of thin specimens, occurs largely by a shear mechanism.
2. In the Fe-16Cr-12Ni alloy the reversed γ has an almost identical crystallographic orientation to the original (retained) γ. Individual complete grains of α' appear to undergo reversion to produce corresponding grains of reversed γ. In the carbon-containing alloy there is evidence that each α' grain produces several regions of reversed γ which are misoriented with respect to each other.
3. Dilatometric measurements indicate that with relatively slow heating rates some thermally activated transformation occurs in addition to reversion by shear.
4. The increase in strength of austenite achieved by reversion is associated with the high dislocation density of the reversed γ regions and the presence of stacking faults and twins.

Acknowledgements

Acknowledgements are made to Professor J. G. Ball for the provision of research facilities; to the Science Research Council and to the Ministry of Defence (formerly Ministry of Technology) for support of the work; to International Nickel Ltd for preparing alloys; to Murex Ltd for supplying chromium and to Dr T. H. Coleman for helpful discussions. The results have formed part of a thesis presented by one of the authors (H.S.) for the Ph.D. Degree of London University.

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Received 5 March and accepted 22 March 1973.