Permanent elimination of the yield-point phenomenon in AISI 430 stainless steel by skin-pass rolling

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The different yielding behaviour of skin-pass and skin-pass plus annealed samples of AISI 430 stainless steel and its dependence on substructure has been studied. Skin-pass specimens show no yield elongation in tensile testing and do not strain age at room temperature. According to TEM observations, this seems to be due to submicroscopic precipitation of low-carbon carbonitrides – most probably Cr_2 (C,N) – preferably on grain boundaries. The locking of interstitials (N, C) by these precipitates could explain the absence of discontinuous yielding. On the other hand, annealing at 700 °C of skin-pass samples dissolves the carbonitride precipitates, interstitial solutes are able to segregate on dislocations, and the pinned dislocations give rise to yield-point phenomena.

1. Introduction

Ferritic stainless steels (SS) of 17% Cr are frequently shaped at room temperature by sheet-pressing processes, such as deep-drawing. In comparison with the austenitic series, they offer the advantage of having a lower strain-hardening capacity. This characteristic of ferritic SS is very convenient for cold-working operations, because, to reach the same degree of deformation, smaller stresses are required and tool and die wear is decreased.

Nevertheless, annealed ferritic SS show a yield point, i.e. a heterogeneous transition from elastic to plastic deformation in the stress–strain curve resulting from a uniaxial tension test. The yield-point phenomenon is well known in annealed low-carbon steel. If such a steel is tested in tension, the stress rises as the sample is strained in the elastic zone, and suddenly falls when the first yielding occurs. After this initial drop, the stress fluctuates about some fairly constant value, while elongation continues, and then begins to rise again (work hardening). The elongation which occurs at about constant load is called yield elongation, yield-point elongation or Lüders strain.

Yield-point behaviour of low-carbon steel is usually associated with the presence of small amounts of solute interstitial atoms. The explanation of this phenomenon was the earlier great success of the dislocation theory [1]. Interstitial carbon and nitrogen atoms are very mobile in b c c ferrite, even at room temperature. They migrate to the space provided by dislocations, segregating in them (Cottrell atmospheres). The elastic interaction dislocation–impurity atmosphere is quite large, so that dislocations are strongly pinned. No dislocations are available for easy glide and a high stress must be applied either to pull dislocation lines free from their segregated atoms or to generate new dislocations from points of stress concentration; then slip can occur at a lower constant stress. In tension testing, if the yielded sample is unloaded and reloaded shortly thereafter, no yield point is observed, because enough unpinned dislocations are present. However, if the mild-steel specimen rests for a few days at room temperature, so that interstitials migrate to the dislocations once more, the yield point returns on reloading [2–4]. This new phenomenon is called static strain ageing.

A negative practical consequence of yield elongation is the appearance of surface irregularities on the deformed sample, known as Lüders bands, Hartmann lines, Piobert effect and stretcher strains. Stretcher strain markings disfigure the steel. To prevent them, sheets intended for deep-drawing applications are lightly prerolled [5] and used before ageing. This skin-pass or temper-rolling operation produces sufficient fresh dislocations to allow a subsequent continuous plastic flow.

Annealed ferritic SS sheets are also subjected to a skin-pass rolling of about 1% reduction in thickness. This small degree of deformation is able to suppress permanently the yield elongation and the formation of undesirable markings, because, contrary to skin-passed mild steels, ferritic SS do not age at room temperature [6]. In addition, skin-passing improves the hardness, flatness and surface finish of sheets.

The purpose of this work was to try to explain the permanent elimination of the yield elongation of 17% Cr ferritic SS by skin-passing. The substructures of samples both in the skin-pass and the annealed

TABLE I Chemical composition (wt %) of AISI 430 SS

C	Si	Mn	S	P	Cr	Ni	Mo	Cu	Sn	N	Fe
0.043	0.43	0.41	0.002	0.039	16.38	0.18	0.01	0.04	0.08	0.024	Bal.

condition were revealed by TEM and the different yielding behaviour is discussed in terms of the TEM observations.

2. Experimental procedure

2.1. Materials

The material used in this investigation was a commercial AISI 430 SS sheet, 2 mm thick and 2B finish (cold-rolling, annealing, pickling and skin-passing). The chemical composition of this steel is shown in Table I.

Some samples of the steel in the as-received state (skin-pass) were annealed in liquid lead (700 °C, 30 min) and air-cooled. Other samples were cold-rolled 50% and annealed at 700 °C for 30 min, followed by air-cooling.

Figure 1 Optical micrograph showing the microstructure of AISI 430 steel in the as-received (skin-pass) condition (parallel to the rolling plane).

2.2. Microscopy

Structural studies were carried out by optical microscopy and, specially, by TEM. For optical microscopy examination, the polished specimens were subsequently electropolished and finally etched electrolytically at 2.5 V for 4 s in a solution of 20% HCl in methanol.

TEM specimens were prepared from 3 mm diameter discs. The samples, taken parallel to the rolling plane, were first thinned chemically (50% HCl, 5% H_3PO_4, 10% HNO_3, 35% H_2O) and then mechanically down to about 0.12 mm thick. Thin-film regions for electron microscopy were then obtained by double-jet electropolishing of the 3 mm discs using a 5% $HClO_4$ aqueous solution at 10 °C. Electropolishing was performed at 23 V with a current of 55 mA. The foils were examined in a Philips CM-12 microscope, operating at 100 kV, and in a Jeol 200, operating at 120 kV. Both bright-field and electron diffraction modes of operation were employed.

Grain and particle mean sizes were measured by image-analysis techniques.

2.3. Tension tests

Both skin-pass and annealed samples were tension tested at room temperature in an Instron 500 kN universal testing machine, using a load rate of 0.2 kN s^{-1}. Flat test specimens (dimensions 250 mm × 13.2mm × 2mm) were prepared in accordance with the Spanish standard UNE 36–401–81. The longitudinal dimension corresponds to the rolling direction.

3. Results and discussion

3.1. Optical microscopy

The microstructure of AISI 430 SS, in the as-received condition, is shown in Fig. 1. The micrograph was taken parallel to the rolling plane of the sheet. The structure consists of carbide particles dispersed in a matrix of ferrite. Electron diffraction (see next section) reveals that these particles are $M_{23}C_6$ mixed carbides. The distribution of carbides (mean size = 0.71 μm, average interparticle spacing = 7.9 μm) is not homogeneous. Observations in the longitudinal cross-section of samples show some alignment of carbides in the rolling direction, as result of the thermo-mechanical history. On the other hand, the degree of deformation caused by skin-passing is so small (1%), that no other structural feature is detected by optical microscopy. In ferrite, of which the average grain size is 16 μm, it must be taken into account that it contains a high amount of chromium in substitutional solid solution.

3.2. Electron microscopy

3.2.1. Skin-pass structure

A general view of the as-received structure, consisting of polygonal ferrite grains, large carbides and dislocations, is shown in Fig. 2. Dislocations tend to group in the neighbourhood of grain boundaries. However, the dislocation substructure is not uniform. Long, straight dislocations are frequently observed in regions of the interior of grains. Sometimes, the dislocations have interacted at the points of intersection (Fig. 3). Electron diffraction proves that the large particles in Figs 1 and 2 are f c c (face-centred cubic) $M_{23}C_6$ carbides (Fig. 4). The measured lattice parameter is a = 1.046 nm. A lattice constant of a = 1.066 nm has been found for $Cr_{23}C_6$ [7]. This carbide dissolves very few chromium nitrides [8].

An important feature of the skin-pass steel structure is the presence of submicroscopic precipitates, mainly

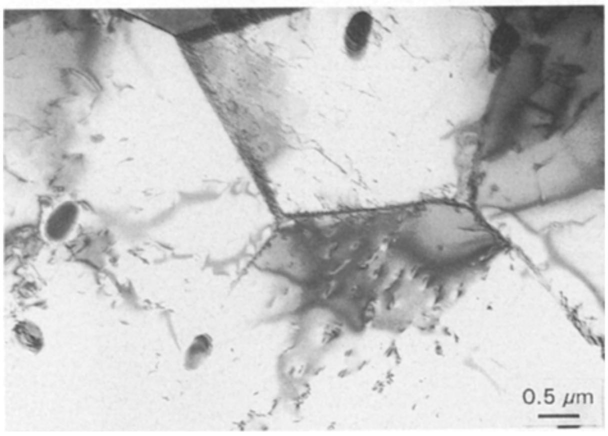

Figure 2 $M_{23}C_6$ carbides and dislocations grouped in the neighbourhood of grain boundaries. Skin-pass steel.

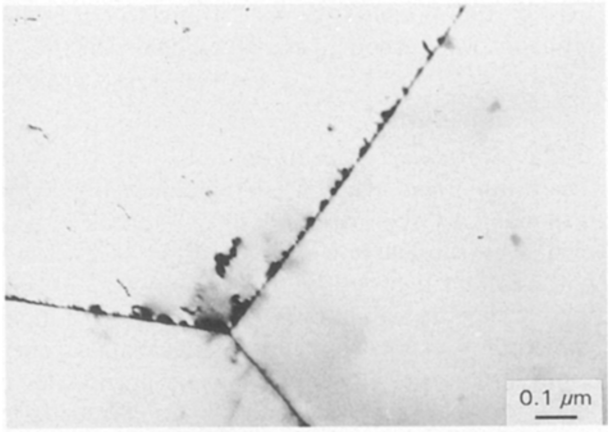

Figure 5 Submicroscopic precipitates on grain boundaries in skin-pass steel.

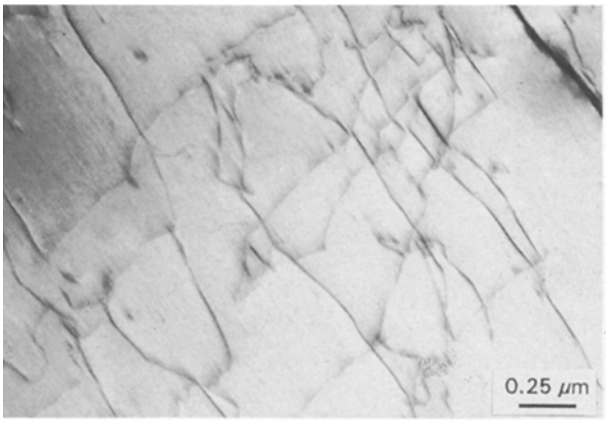

Figure 3 An aspect of the dislocation structure of the skin-pass steel. Straight dislocations have interacted forming a cross-grid.

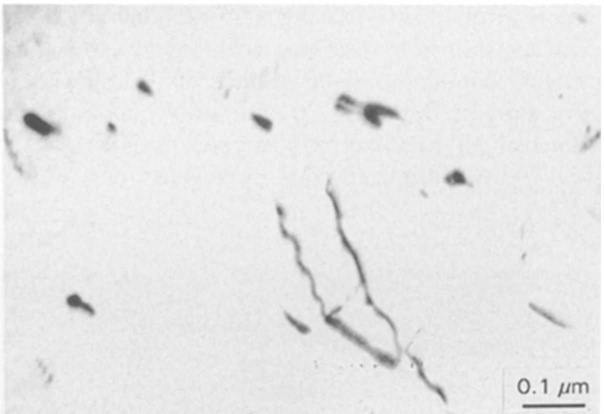

Figure 6 Submicroscopic precipitates inside the grains in skin-pass steel. They are not associated with dislocations.

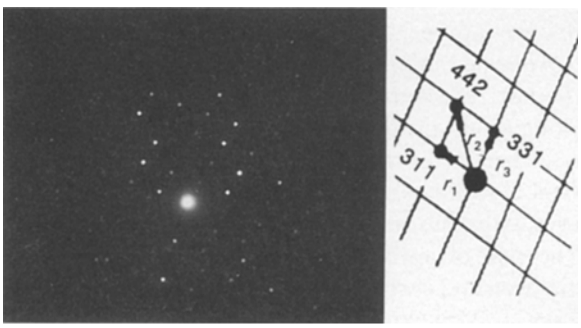

Figure 4 Electron diffraction pattern of particles in Fig. 1.

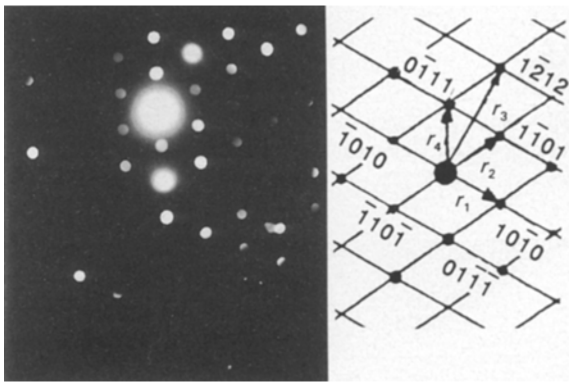

Figure 7 Electron diffraction pattern of submicroscopic precipitates which appear in Figs 5 and 6. Skin-pass steel.

at grain boundaries (Fig. 5). They are also observed inside the grains, usually not associated with dislocations (Fig. 6).

The size of these particles is smaller than 0.05 µm. The electron diffraction pattern of such precipitates (Fig. 7) corresponds to an h c p (hexagonal closed-packed) crystal [9], the *a* parameter being $a = 0.478$ nm. This value of *a* is in good agreement with the lattice parameters of the hexagonal structure of Cr_2N ($a = 0.478$–0.481 nm, $c = 0.444$–0.448 nm) determined by X-ray diffraction by other authors [10, 11] Furthermore, Imai *et al.* [12], in their study of the Cr–Fe–N system, predict the existence of Cr_2N at room temperature.

On the other hand, the $Cr_2(C, N)$ carbonitride, up to 4.5%C, also has a hexagonal structure with lattice parameters $a = 0.483$–0.485 nm and $c = 0.448$–0.445 nm [13]. Therefore, the submicroscopic precipitates of

Figs 5 and 6 could be Cr_2N nitrides or, more probably, low-carbon $Cr_2(C, N)$ carbonitrides.

3.2.2. Annealed structures
The transmission electron micrograph of Fig. 8 corresponds to a skin-pass and 700 °C annealed specimen. The substructure consists of $M_{23}C_6$ carbide particles and a group of dislocations near a grain boundary. The dislocations have adopted a polygonized, low-energy configuration, recalling a honeycomb structure. The submicroscopic precipitates of $Cr_2(C, N)$ are not present, either in grain boundaries or inside the grains (Fig. 9). The annealing treatment at 700 °C has dissolved them.

These observations seem to indicate that the precipitation of $Cr_2(C, N)$ carbonitrides in the skin-pass samples is strain-induced. To confirm this, samples in a structural condition similar to that of commercial sheets prior to skin-passing were examined by TEM. The pre-skin-pass state was achieved by laboratory-process simulation (cold rolling 50% followed by annealing at 700 °C for 30 min and air cooling). The structure of pre-skin-pass samples is illustrated in Fig. 10. In addition to grain boundaries, only $M_{23}C_6$ carbides and a low-density of dislocations are present. Carbonitride precipitates are not detected. Referring to dislocations, the substructure in Fig. 10 results from recrystallization, while that shown in Fig. 9 corresponds to a recovery process. The small amount of cold working (1%) produced by the skin-pass operation (subcritical plastic deformation) is unable to lead to recrystallization after annealing.

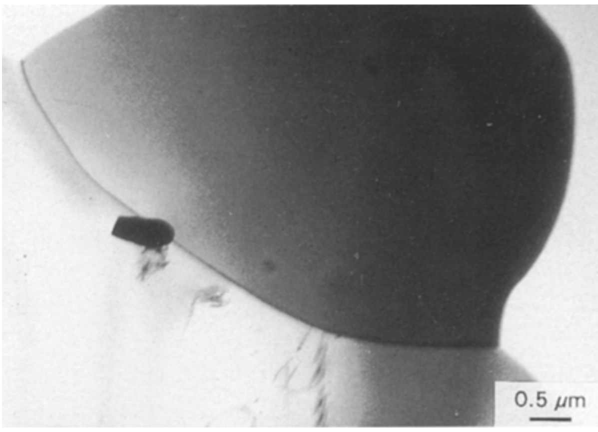

Figure 10 Typical recrystallized TEM structure of pre-skin-pass steel. The pre-skin-pass state was achieved by process simulation.

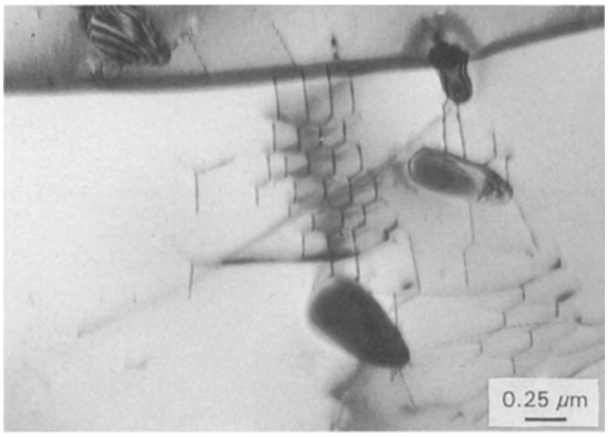

Figure 8 $M_{23}C_6$ carbides and polygonized dislocations near a grain boundary. Skin-pass and 700 °C annealed specimen.

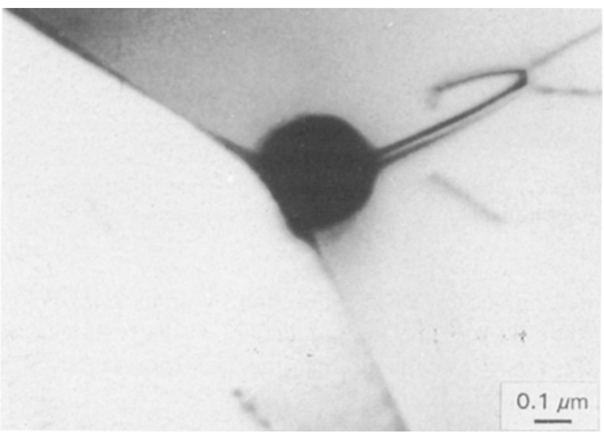

Figure 9 No submicroscopic precipitates are present either in grain boundaries or inside the grains, in skin-pass plus annealed steel. Compare this TEM structure with those shown in Figs 5 and 6.

3.3. Tension tests
The stress–strain curve resulting from tension testing a skin-pass sample at room temperature is shown in Fig. 11. The sample was tested more than 2 years after skin-pass rolling. This curve displays with increasing stress a continuous, gradual departure from elastic to plastic behaviour. The skin-pass ferritic SS has not strain-aged. On the other hand, the engineering stress–strain diagrams of skin-pass and annealed tensile-test specimens exhibit a rather abrupt plastic yielding, the initial increase of strain occurring without any appreciable increase of stress (Fig. 12).

Annealed mild steel has a tensile behaviour similar to that reflected in Fig. 12. To eliminate the yield elongation, in order to avoid the appearance of stretcher strain markings in shaping operations, mild steel is skin-pass rolled [5]. But skin-pass mild steel ages with time and the discontinuous yielding returns. The rate of ageing is controlled by the diffusion of nitrogen and carbon atoms [14]. It usually takes a few days at room temperature. However, skin-pass ferritic SS does not age, as stated by Randak [6] and shown in Fig. 11 for a sample tested more than 2 years after the skin-pass operation.

TEM was used to investigate the substructures of skin-pass and skin-pass plus annealed specimens of AISI 430 SS: skin-pass samples showed strain-induced precipitation of $Cr_2(C, N)$ carbonitrides (Figs 5 and 6), mainly in grain boundaries, while no such precipitates were present in specimens which had been skin-pass and annealed at 700 °C (Figs 8 and 9). These structural observations combined with the Cottrell theory of yield-point [1] could explain the different tensile stress–strain behaviour, which appears in Figs 10 and 11.

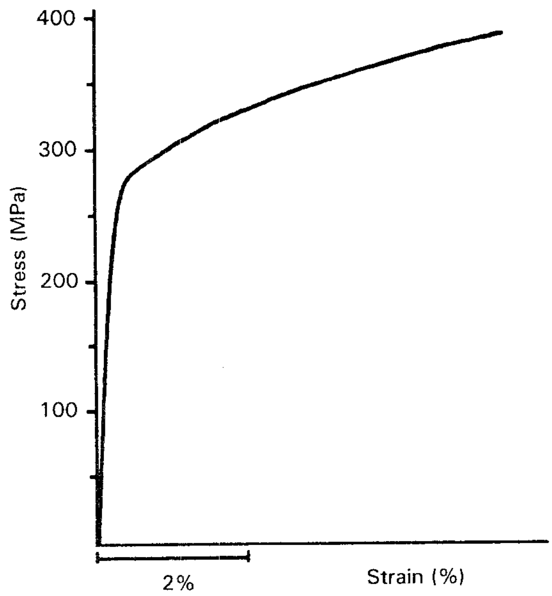

Figure 11 Stress–strain curve of skin-pass AISI 430 SS.

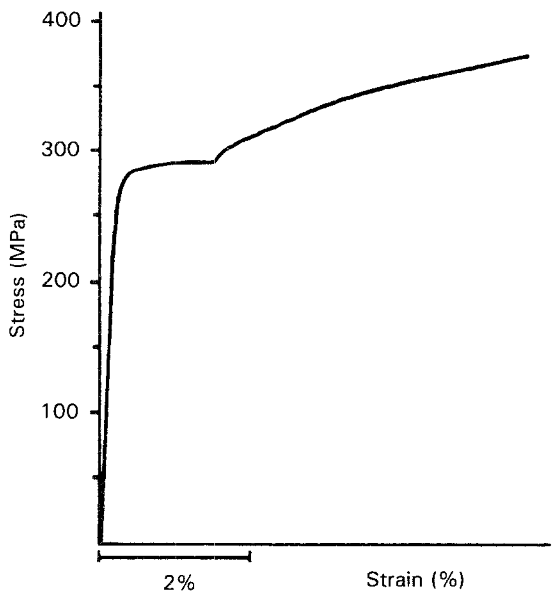

Figure 12 Stress–strain diagram, showing yield elongation, of skin-pass plus annealed AISI 430 SS.

In skin-pass AISI 430 SS, carbon and nitrogen atoms have been taken out of solution in the form of $M_{23}C_6$ carbides and $Cr_2(C, N)$ low-carbon carbonitrides (Figs 2 and 5). However, the small, glide-restricting carbonitride precipitates are not associated with dislocations (Figs 5 and 6), so that enough free dislocations are available for slip. Under this condition, plastic deformation occurs in a smooth, homogeneous manner (Fig. 11). The mechanism operating to avoid yield-point phenomena is, in a certain way, similar to that used in ordinary mild steel when some aluminium, vanadium or titanium is added to fix the nitrogen as nitride precipitates [15].

On the other hand, skin-pass and annealed specimens contain nitrogen and a few carbon atoms in solid solution in ferrite, because carbonitrides have dissolved (Figs 8 and 9). Solute nitrogen and carbon atoms segregate on dislocations, forming Cottrell atmospheres [1]. Dislocations are thus pinned by interstitial interactions (impurity locking). This anchoring effect against the onset of plastic deformation produces heterogeneous yielding (yield-point elongation), as reflected in Fig. 12.

4. Conclusion

Skin-pass rolling, as a final step in the manufacture of AISI 430 SS sheets, brings about the permanent elimination of yield-point phenomena. This effect is not produced in ordinary mild steels, where the elimination caused by the skin-pass operation has only a transitory character, because such steels strain age at room temperature. AISI 430 SS shows, nevertheless, a discontinuous yielding behaviour in tensile testing, when skin-pass specimens are subjected to an annealing treatment at 700 °C for 30 min, followed by air cooling.

The plastic deformation introduced by skin-passing- (about 1% reduction in thickness) gives rise to a strain-induced precipitation of submicroscopic particles, mainly on grain boundaries. These particles have a size smaller than 0.05 μm. The electron diffraction pattern of these fine precipitates corresponds to a h c p crystal structure, the *a* parameter being 0.478 nm. The precipitates could probably be low-carbon carbonitrides of the type $Cr_2(C, N)$. These carbonitrides strongly bond nitrogen and carbon atoms, while a good amount of dislocations remain free to move.

On the other hand, annealing at 700 °C causes dissolution of the carbonitrides, according to TEM observations. This could explain, in the light of the Cottrell theory, the different yielding behaviour of skin-pass and skin-pass plus annealed samples of AISI 430 SS.

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