

Influence of additional elements (Mo, Nb, Ta and B) on the mechanical properties of high-manganese dual-phase steels

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The influence of a small amount of alloying elements such as molybdenum, niobium, tantalum and boron to the 3 wt% Mn ferritic dual-phase steels was examined. These elements give a microstructure in which the fine martensite phase is well dispersed in the ferrite matrix, which leads to a remarkable reinforcement effect on mechanical properties. It is possible to obtain a tensile strength, σ_R , approaching 700 MPa with a fracture elongation, A_R , of more than 40%, or $\sigma_R \sim 1000$ MPa with $A_R = \pm 20\%$ with relatively simple heat treatment.

1. Introduction

In our previous works [1], the structure and mechanical properties of low-carbon and high-manganese (about 3 wt% Mn) ferritic dual-phase steels were examined as a function of heat treatments. It was observed that, because of the high manganese content, these alloys can be easily prepared even by a simple air cooling and they consist of a fine dispersion of the martensitic phase which leads to high strength and good ductility. The addition of a small amount of silicon further increases the tensile strength without a significant loss of formability.

On the other hand, high-strength low-alloy (HSLA) steels, precursors of ferritic dual-phase steels, were developed from low-carbon steels by combining a small amount of refractory metal elements such as vanadium, molybdenum, niobium or titanium [2]. HSLA steels represent high strength, derived from grain refinement and precipitation strengthening due to minor additional elements but they are less formable than low-carbon steels, while the dual-phase steels have better formability than HSLA steels of similar strength. Consequently, it may be expected that some beneficial effects may be obtained by the addition of alloying elements to the dual-phase steels. Thus we hope to improve further the mechanical properties of dual-phase steels: i.e. higher strength without too much loss of formability in comparison with the high-manganese dual-phase steels developed in our previous work [1].

The basic chemical composition of the steels prepared in this work was 0.1% carbon, 3% manganese and 0.5% silicon (wt %), which gave the most promising mechanical properties in our previous work. Then, we first chose as minor alloying elements, molybdenum, niobium and tantalum. We also tried a boron addition because this element is used in high-strength steels, but it is expected to show different effects from the previous group of refractory metal

elements on mechanical properties, especially in the case of long heat treatment.

2. Experimental procedure

The alloy specimens were prepared in a similar way to that described in our previous paper [1]. The mixture of raw materials was melted by induction and cast into a water-cooled mould under an argon atmosphere. Homogenization treatment of the cast ingots was carried out by heating at 1100°C for 20 h under argon flow. Then the ingots were hot- and cold-rolled from 20 to 4 mm thickness. Flat tensile specimens of 32 mm effective length, 6.25 mm width and 3 mm thick were machined prior to heat treatments.

Table I shows the results of chemical analysis carried out on the prepared alloy steels.

We measured the transformation temperatures A_{c1} , A_{c3} , A_{r3} and A_{r1} of the prepared steels by the dilatometric method. The alloy specimens were heated in vacuum from room temperature up to about 900°C and then cooled with a constant heating and cooling rate of 300°C h⁻¹. The results obtained from this heat-treatment cycle are summarized in Table II for each kind of steel.

The mechanical tensile tests were carried out using an Instron machine at room temperature using a 25 mm gauge length extensometer with a cross-head speed of 2 mm/min⁻¹. The 0.2% offset yield strength, σ_E (MPa), the ultimate tensile strength, σ_R (MPa), the

TABLE I Compositions of materials (wt %)

Alloy No.	C	Mn	Si	Mo	Nb	Ta	B	Fe
1	0.13	2.88	0.56	0.10	—	—	—	bal.
2	0.15	2.89	0.56	—	0.10	—	—	bal.
3	0.18	2.88	0.54	—	—	0.09	—	bal.
4	0.09	3.12	0.57	—	—	—	0.025	bal.
5	0.15	3.01	0.51	0.10	—	—	—	bal.
6	0.11	2.89	—	—	—	—	—	bal.
7	0.08	3.07	0.23	—	—	—	—	bal.

TABLE II Transformation temperatures A_{c1} , A_{c3} , A_{r3} and A_{r1} ($^{\circ}\text{C}$)

Alloy No.	A_{c1}	A_{c3}	A_{r3}	A_{r1}
1	653	840	606	374
2	676	851	699	387
3	664	832	695	361
4	629	844	614	420
6	681	811	653	370
7	656	822	664	399

uniform and fracture elongation, A_u and A_R (%), were measured on engineering stress-strain diagrams.

Strain hardening behaviour can be evaluated by the coefficient n obtained from the following Hollomon's relation

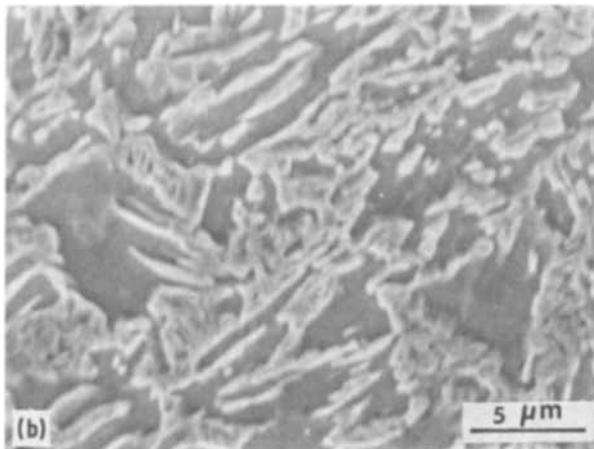
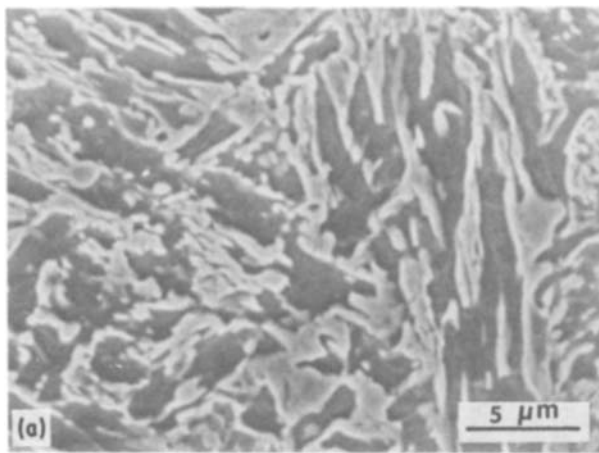
$$\sigma = a\varepsilon^n$$

where σ is a true stress and ε a true plastic strain.

Vickers microhardness was measured on polished surfaces with a weight of 50 to 100 g. The microstructures were examined by scanning and transmission electron microscopes, SEM and TEM.

3. Experimental results

In the first series of this work, we examined the influence of the heat-treatment temperatures on four kinds of steels, 1, 2, 3 and 4 in Tables I and II. The specimens were heated at three different temperatures, 700, 750 and 800 $^{\circ}\text{C}$, respectively, for 10 min and then cooled in air to room temperature. These heat treatments were carried out using a lead bath.



In the second series, more detailed experiments were carried out on alloy 5, molybdenum containing steel as well as on alloys 6 and 7.

3.1. Structure and mechanical properties of steels, heat treated at 700 to 800 $^{\circ}\text{C}$ and air-cooled

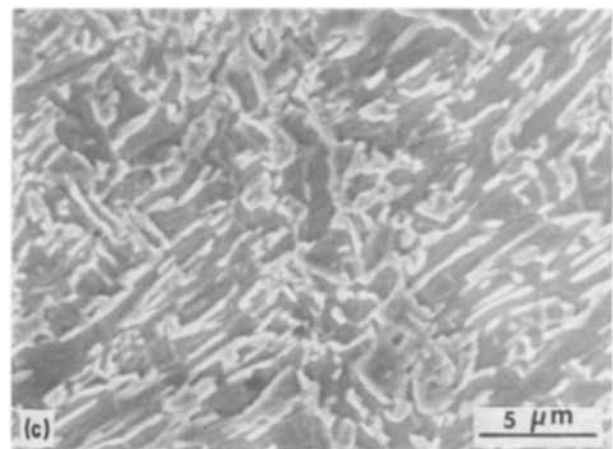
Fig. 1 shows some examples of microstructures observed by SEM on steel 1, prepared by heating for 10 min at 700, 750 and 800 $^{\circ}\text{C}$, respectively. The martensite phase is formed as fine needles or particles and dispersed in the ferrite matrix. There is no appreciable difference between the four kinds of steels prepared, nos 1 to 4, but it is to be noted that these structures are moreover finer than those of the Fe-0.1% C-3% Mn steels reported in our previous paper [1].

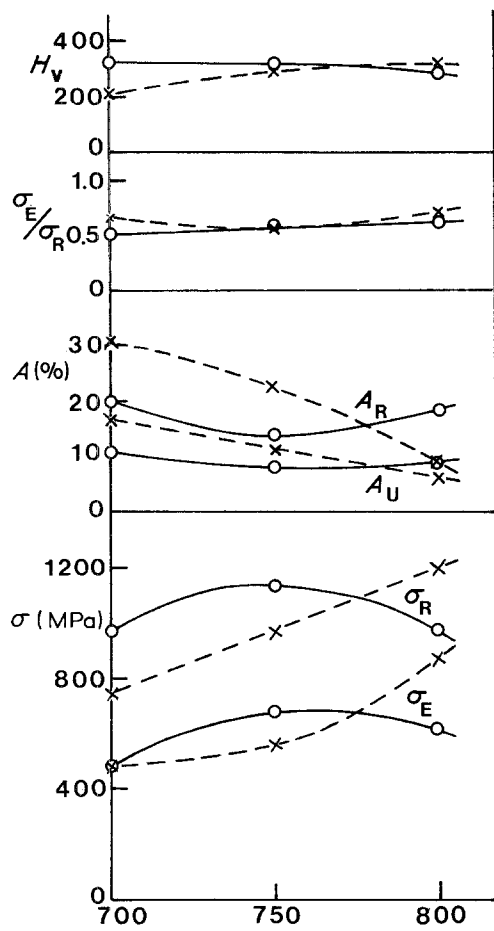
X-ray diffraction analysis with a goniometer recording shows the presence of retained austenite phase in a small but not negligible amount. In reality, the volume fraction of retained austenite, estimated by X-ray diffraction is nearly 7 to 10%, 1 to 2% and 3 to 4% for the steels prepared by heat-treatment at 700, 750 and 800 $^{\circ}\text{C}$, respectively.

Fig. 2 summarizes the results of mechanical tests on the four groups of steels, prepared by air-cooling, as a function of annealing temperature: 0.2% offset yield- and ultimate-tensile strength, σ_E and σ_R , uniform- and fracture-elongation, A_u and A_R , σ_E/σ_R ratio and Vickers microhardness, H_v . The strain hardening coefficient, n , is nearly 0.22 ± 0.04 for all specimens and there is no appreciable difference depending on the kinds of steels or annealing temperature.

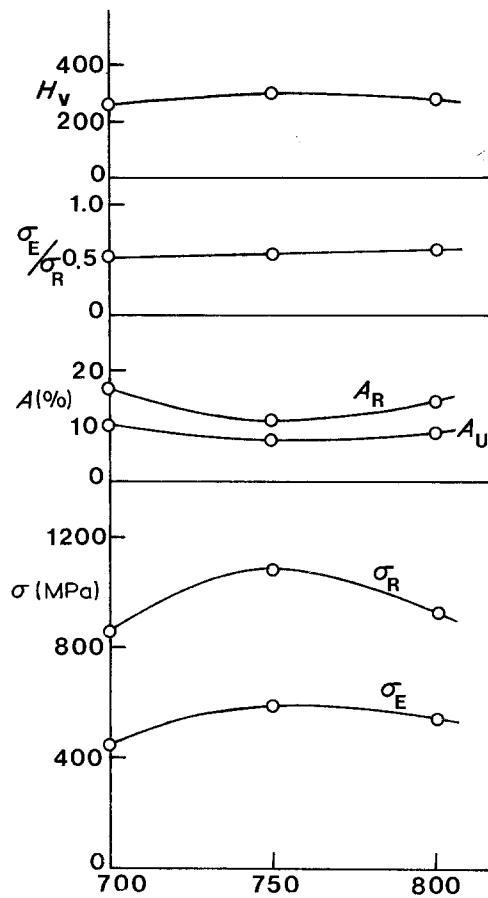
We also performed tensile tests on steel specimens prepared by heat treatment at 700 $^{\circ}\text{C}$ for 10 min, followed by quenching in water at room temperature. The results are summarized in Table III. Contrary to our expectation, these water-quenched specimens from 700 $^{\circ}\text{C}$ show lower mechanical strength and larger formability than the alloys prepared by air cooling from the same temperature, and this tendency is visible more clearly for the molybdenum-, niobium-

Figure 1 Structure of the Fe-0.13% C-2.88% Mn-0.56% Si-0.10% Mo steels, observed by scanning electron microscopy (SEM): (a) annealed at 700 $^{\circ}\text{C}$, (b) 750 $^{\circ}\text{C}$, and (c) 800 $^{\circ}\text{C}$, for 10 min and cooled in air.

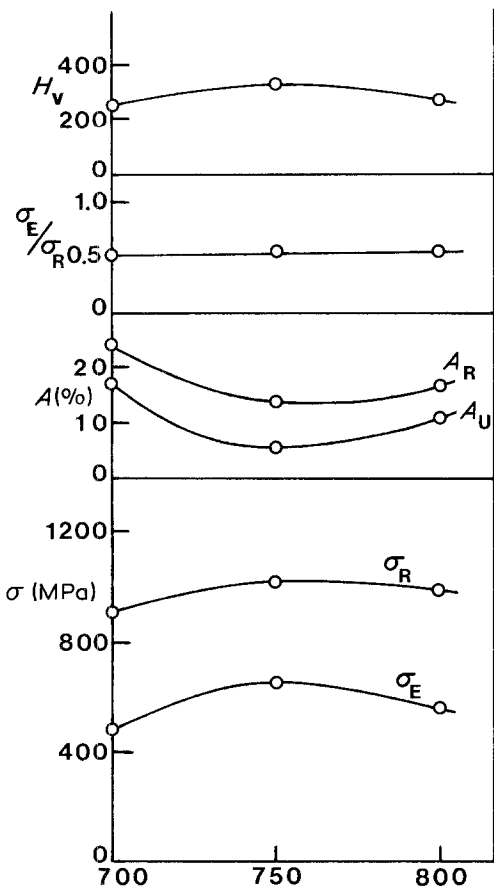




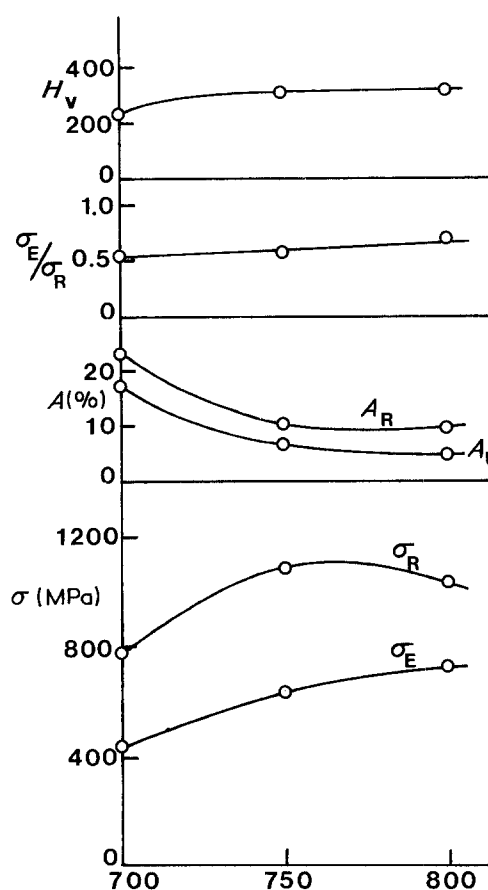
(a) ANNEALING TEMPERATURE(°C)



(b) ANNEALING TEMPERATURE(°C)



(c) ANNEALING TEMPERATURE(°C)



(d) ANNEALING TEMPERATURE(°C)

Figure 2 (—) The results of mechanical tests on the air-cooled specimens after 10 min annealing at high temperatures 700, 750 and 800°C. (a) Fe-0.13% C-2.88% Mn-0.56% Si-0.10% Mo, (b) Fe-0.15% C-2.89% Mn-0.56% Si-0.10% Nb, (c) Fe-0.18% C-2.88% Mn-0.54% Si-0.09% Ta, (d) Fe-0.09% C-3.12% Mn-0.57% Si-0.025% B. (a) (---) The results of mechanical tests on steel 5 (Fe-0.15% C-3.01% Mn-0.51% Si-0.10% Mo), prepared by annealing for 10 min followed by water-quenching.

TABLE III Experimental results obtained on steels, prepared by water quenching after annealing for 10 min at 700° C

Steel No.	σ_E (MPa)	σ_R (MPa)	A_u (%)	A_R (%)	σ_E/σ_R	H_v
1	558	758	15.9	29.2	0.74	273
2	521	768	13.1	23.3	0.68	264
3	542	793	15.0	25.5	0.68	286
4	521	722	12.4	21.7	0.72	253

and tantalum-bearing steels. We will discuss these results later.

3.2. Fe-0.15% C-3.01% Mn-0.51% Si-0.10% Mo steels, heat-treated at 700 to 800° C and water-quenched

The broken-line curves in Fig. 2a show the results of mechanical tests on the molybdenum-containing steel, No. 5, prepared by annealing for 10 min at 700, 750 and 800° C, respectively, followed by quenching in water at room temperature.

The alloy specimens become stronger mechanically and less ductile as the annealing temperature increases because of the increase in the amount of martensite phase. The strain hardening coefficient, n , is nearly 0.2 for all of these water-quenched steels. The volume fractions of retained austenite, estimated by X-ray diffraction are about 5, 3 and 2% for three different annealing temperatures, 700, 750 and 800° C, respectively.

It is observed once again, in comparison with the results expressed by the full lines in Fig. 2a, that the steel specimens prepared by water-quenching after annealing for 10 min at 700 or 750° C show higher uniform and fracture elongations and lower mech-

anical strength than those prepared by a simple air-cooling on steel 1, even though there is some small difference of chemical composition between these two kinds of steels, 1 and 5.

3.3. Effect of tempering

As a next step, we examined the effects of tempering on the mechanical properties of three kinds of dual-phase steels, nos 6, 7 and 5.

Figs 3 and 4 summarize the results on the Fe-0.11% C-2.89% Mn and the Fe-0.08% C-3.07% Mn-0.23% Si steels, respectively, which were prepared by water-quenching after annealing for 10 min at 800° C, followed by tempering at 700° C and air-cooling. In these figures, n represents the strain hardening coefficient and C_γ a volume fraction of retained austenite estimated by X-ray diffraction.

By a short-term (less than 5 min) tempering at 700° C, the mechanical strength decreases rapidly and then it recovers slightly after a long-term heating of more than 10 min and it nearly attains 700 MPa, while the ductility of these steels increases remarkably and their fracture elongation approaches 40% after about 20 min tempering. The strain hardening coefficient, n , and the volume fraction of retained austenite, C_γ , show a similar variation as that of mechanical strength as a function of tempering time. It should be noted that the amount of the retained austenite increases on tempering at 700° C, almost reaching 10%.

Figs 5a to c show the evolution of microstructure of the Fe-0.11% C-2.89% Mn steel, observed in the scanning electron microscope. After a short tempering of less than 5 min at 700° C, the needles of martensite

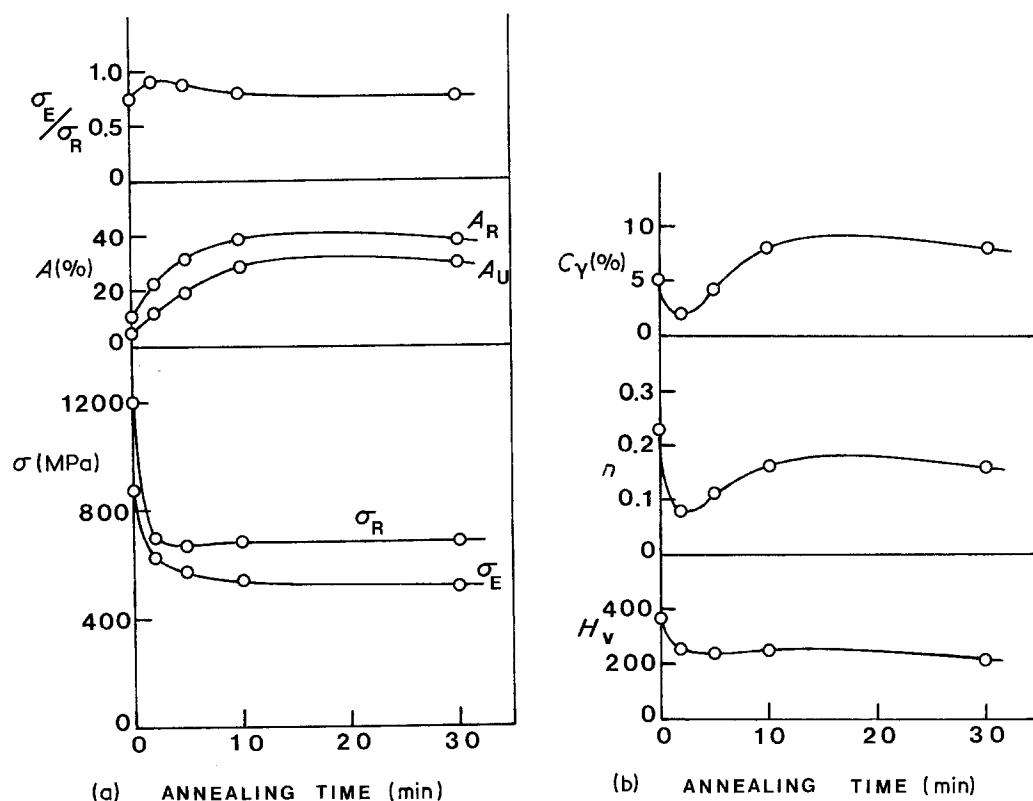


Figure 3 Effect of annealing time on the mechanical properties of the water-quenched Fe-0.11% C-2.89% Mn steel, tempered at 700° C: (a) mechanical strength, σ_E , σ_R ; elongation A_u , A_R and σ_E/σ_R ratio, (b) Vickers microhardness, H_v , strain hardening coefficient, n , and volume fraction of retained austenite, C_γ .

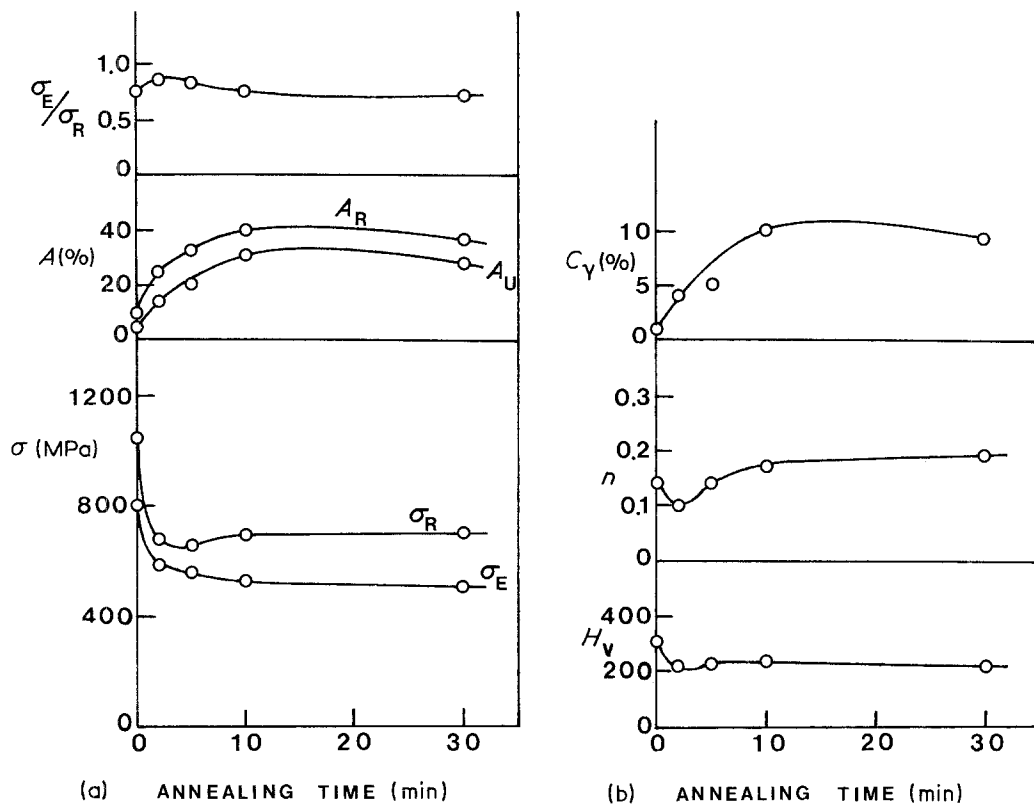


Figure 4 Effect of annealing time on the mechanical properties of the water-quenched Fe-0.08% C-3.07% Mn-0.23% Si steels, tempered at 700°C.

phase formed by annealing at 800°C and water-quenching are broken into small particles which are then rearranged to form a larger equilibrium martensite phase at 700°C. A similar aspect of structural modifi-

cation was also observed on the Fe-0.08% C-3.07% Mn-0.23% Si steel. The softening of these alloys described above coincides with the state of Fig. 5b in which the initial martensite phase is decomposed into small particles.

We also examined the tempering effect in a similar way on the molybdenum-containing steel 5. The alloy specimens, annealed for 10 min at 800°C and quenched in water at room temperature were heated at 700°C for times varying from 2 to 60 min and then air cooled. Fig. 6 summarizes the results obtained on the Fe-0.15% C-3.01% Mn-0.51% Si-0.10% Mo steel. The fracture elongation increases remarkably as the

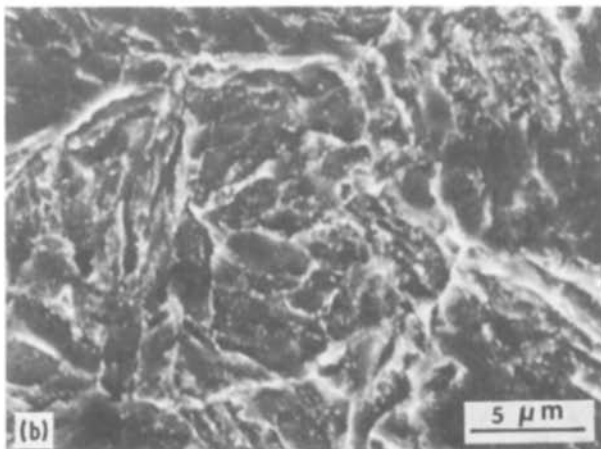
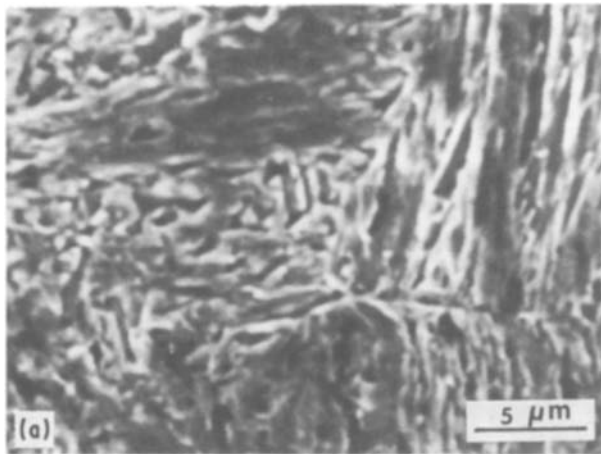
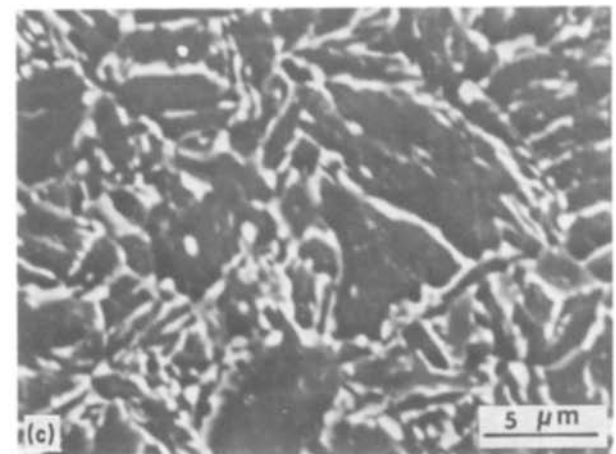


Figure 5 Structural evolution of the Fe-0.11% C-2.89% Mn steel by tempering, observed by SEM: (a) initial state, prepared by annealing for 10 min at 800°C and water-quenching, (b) tempered 2 min at 700°C and air-cooled, (c) tempered 10 min at 700°C and air-cooled.



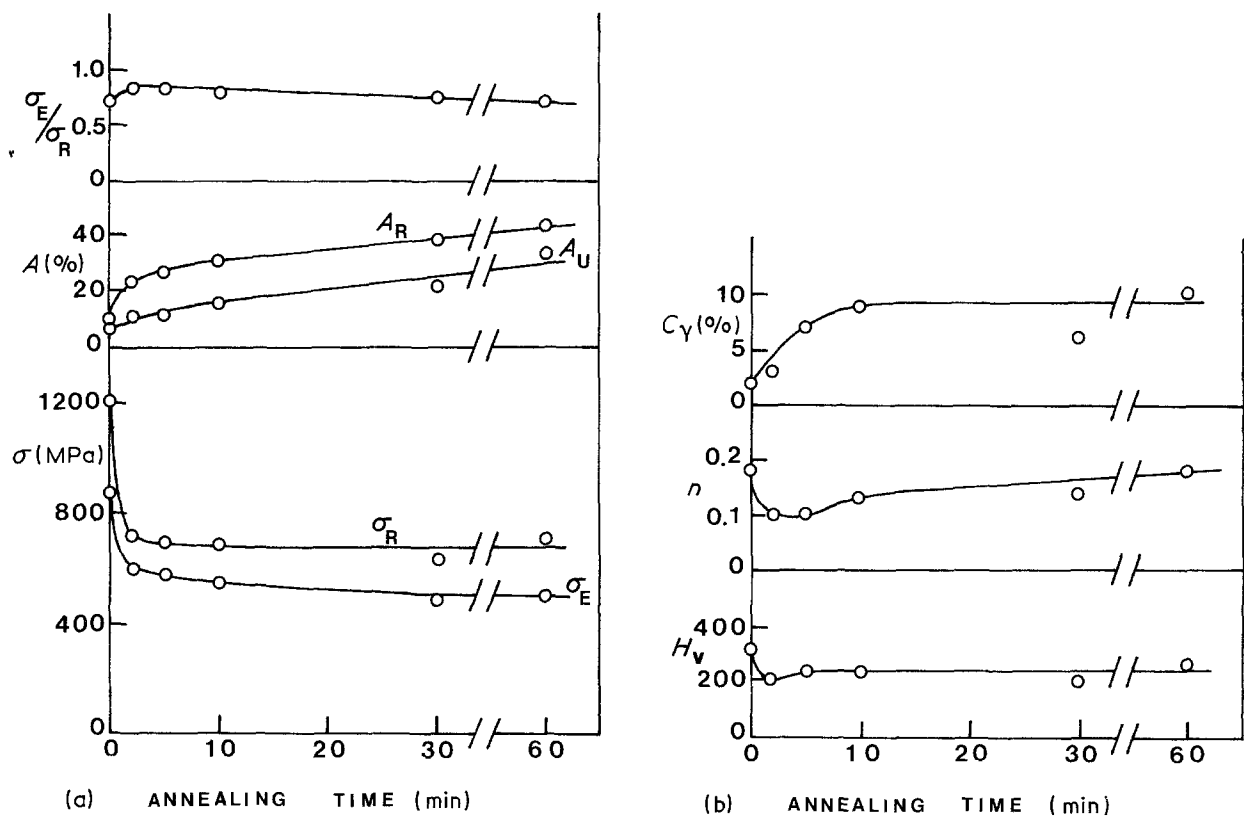


Figure 6 Effect of annealing time on the mechanical properties of the water-quenched Fe-0.15% C-3.01% Mn-0.51% Si-0.10% Mo steel, tempered at 700°C.

tempering time increases and it becomes more than 40% after 60 min heating. Moreover, the mechanical strength decreases rapidly on short time tempering as in the previous cases, but it seems that the molybdenum addition in small amounts restrains this drop of mechanical strength and retains the toughness much more in comparison with the cases of no molybdenum addition. The amount of retained austenite also increases on tempering at 700°C.

Figs 7a to d show the structural evolution of this steel, observed by SEM. Short-term tempering at 700°C decomposes the martensite needle crystals which then coagulate to form larger crystals, but this change of martensite size in the molybdenum-bearing steel is retarded in comparison with the previous cases and it becomes visible after 30 min tempering, as shown in Figs 7b and c. Fig. 7d shows, however, that a number of small precipitates are formed after a long-term tempering.

As a final experiment, we examined the effect of 1 h tempering, as a function of heating temperature, on the same molybdenum-bearing steel, prepared by annealing for 10 min at 800°C and water-quenching. Fig. 8 summarizes these experimental results. The tempering effect on mechanical properties becomes significant at temperatures higher than 600°C.

Figs 9a and b show the structures of this steel after 1 h tempering at 400 and 600°C, respectively. It is observed, by comparing with Figs 7a and d, that fine precipitates are not observed on tempering at 400°C but that they become visible at 600°C.

The observations described above indicate that there are two different mechanisms which modify the mechanical properties of dual-phase steel by

tempering:

1. the softening of ferrite matrix and the decomposition of martensite phase in an initial stage of tempering;

2. the hardening by martensite rearrangement and fine precipitation. Probably, the grain growth by long-term tempering induces the decrease of mechanical strength and fracture elongation, but it seems that this drawback is compensated by precipitation hardening.

All of the steel specimens prepared in this work, even the alloy annealed for 1 h at 400°C, exhibit typical dual-phase steel behaviour in the tensile test, i.e. "continuous" stress-strain curves.

3.4. TEM observation

We examined the microstructure of the dual-phase steels by transmission electron microscopy of thin films prepared by the electrolytical thinning technique using the solution of 5% perchloric acid and 95% acetic acid at 0 to 5°C. We tried especially to identify the retained austenite phase which is expected to exist in the martensite region, but we found that it was fairly difficult to determine its location because the martensite phase which is rich in carbon is thinned much less than the ferrite matrix, and so the martensite phase remains in general as a too thick particle or island in a thinned ferrite matrix, and it falls easily from the thinned specimen at the end of the thinning process.

Fig. 10a was obtained on the Fe-0.15% C-3.01% Mn-0.51% Si-0.10% Mo steel, prepared by annealing for 10 min at 800°C, followed by water quenching and then annealing for 1 h at 700°C. The amount of retained austenite attains the maximum value,

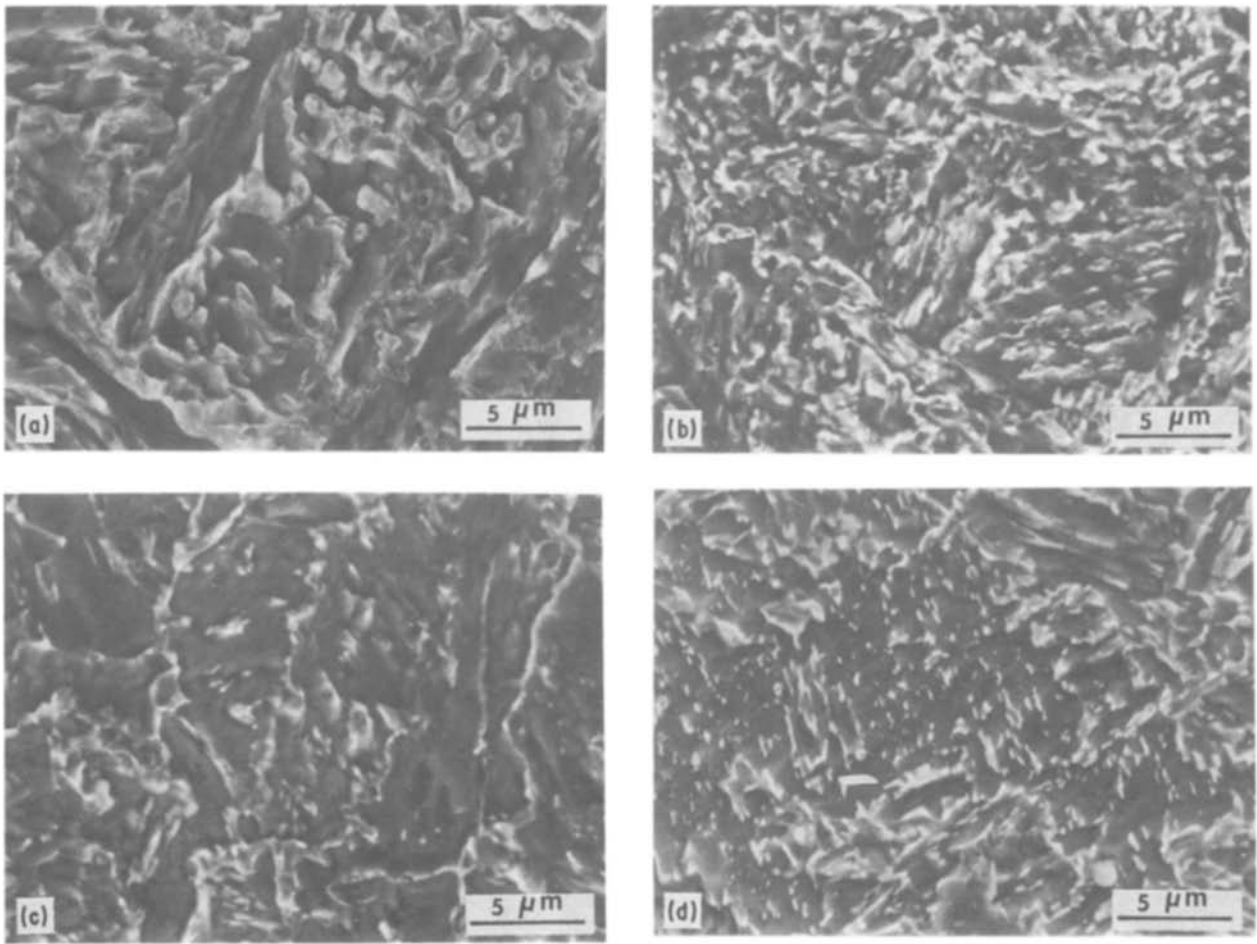


Figure 7 Structural evolution of the Fe-0.15% C-3.01% Mn-0.51% Si-0.10% Mo steel by tempering, observed by SEM: (a) initial state, prepared by annealing for 10 min at 800°C and water-quenched, (b) tempered 5 min at 700°C and air-cooled, (c) tempered 30 min at 700°C and air-cooled, (d) tempered 60 min at 700°C and air-cooled.

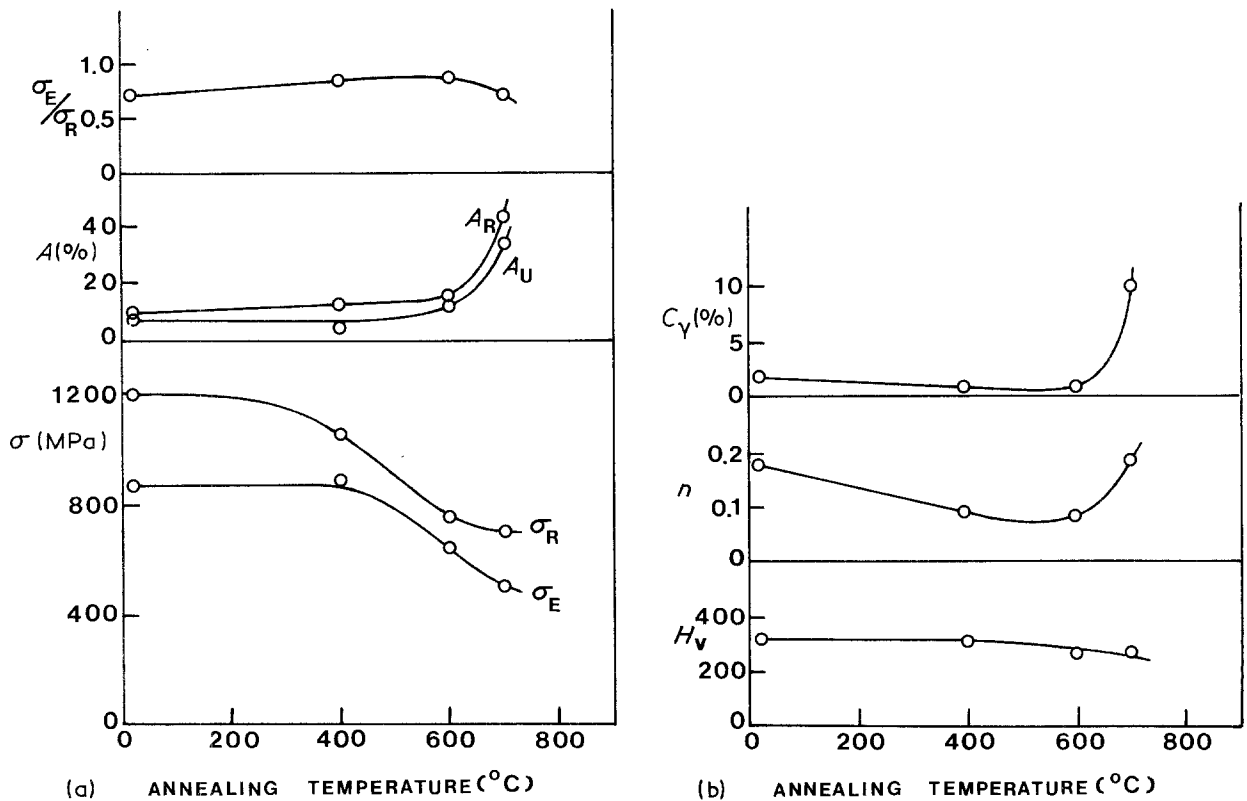


Figure 8 Effect of tempering for 1 h on the mechanical properties of the Fe-0.15% C-3.01% Mn-0.51% Si-0.10% Mo steel, prepared by annealing for 10 min at 800°C followed by water-quenching.

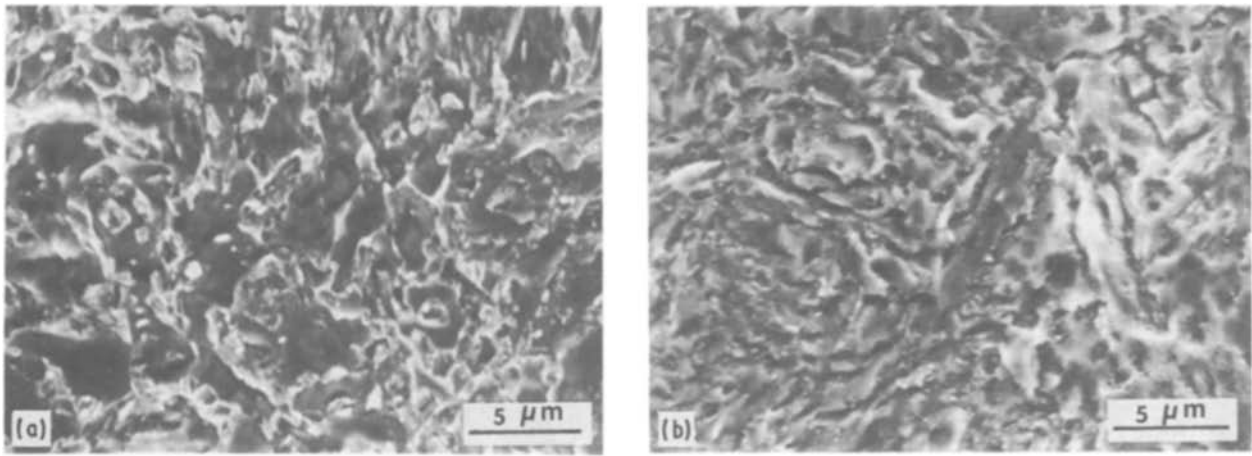


Figure 9 Scanning electron micrographs of the Fe-0.15% C-3.01% Mn-0.51% Si-0.10% Mo steel, prepared by annealing for 10 min at 800°C and water-quenching, after tempering for 1 h at (a) 400°C, and (b) 600°C.

about 10%, in this steel as shown in Fig. 6. Fig. 10a should be compared with Fig. 10b, obtained on the Fe-0.04% C-2.88% Mn steel, prepared by annealing for 10 min at 800°C and then water-quenched, in our previous work [1]. The martensite phase is formed of acicular crystals, showing the typical martensitic morphology.

In Fig. 10a, however, the martensite phase which appears often as an elongated island shows a band structure formed of ferrite and martensite flaky crystals. This band structure is produced by two successive heat treatments, annealing at high temperature (800°C) and tempering at low temperature (700°C). This result indicates that the acicular martensite phase as in Fig. 10b, formed by annealing at high temperature and water-quenching, is decomposed by tempering at lower temperature into the martensite and ferrite lath mixture as shown in Fig. 10a. Matlock *et al.* [3] have also observed by SEM the similar alternative structure of ferrite and martensite acicular crystals, known as Widmanstätten structure, in the dual-phase steel obtained by oil-cooling from high temperature. We observed in a few cases the presence of the retained austenite phase in these band structures by electron diffraction.

4. Conclusion

Our observations in this work show first of all that the addition of a small amount of molybdenum, niobium, tantalum and boron to 3 wt % manganese dual-phase steels gives finer structures than those of the Fe-0.1% C-3% Mn and Fe-0.1% C-3% Mn-Si steels, reported in our previous work [1]. The fine martensite phase is well dispersed in the ferrite matrix and this microstructure leads to the excellent mechanical properties: high strength and large fracture elongation.

In our previous work, we have obtained the ultimate tensile strength $\sigma_R \sim 600$ MPa with the fracture elongation $A_R \sim 30\%$ or $\sigma_R \sim 800$ MPa with $A_R \sim 20\%$ by a simple air cooling from 700°C. We observed in this work that the alloying elements increase the mechanical strength remarkably and we obtained $\sigma_R \sim 1000$ MPa with $A_R \sim 20\%$ on molybdenum- or tantalum-bearing steel prepared by air cooling from 700°C. The boron addition shows a somewhat different effect: the fracture elongation decreases as the annealing temperature increases. This is probably due to the boride precipitation at the grain boundary as observed in boron-bearing steels by high-temperature heating [4].

We observed an unexpected result on alloying

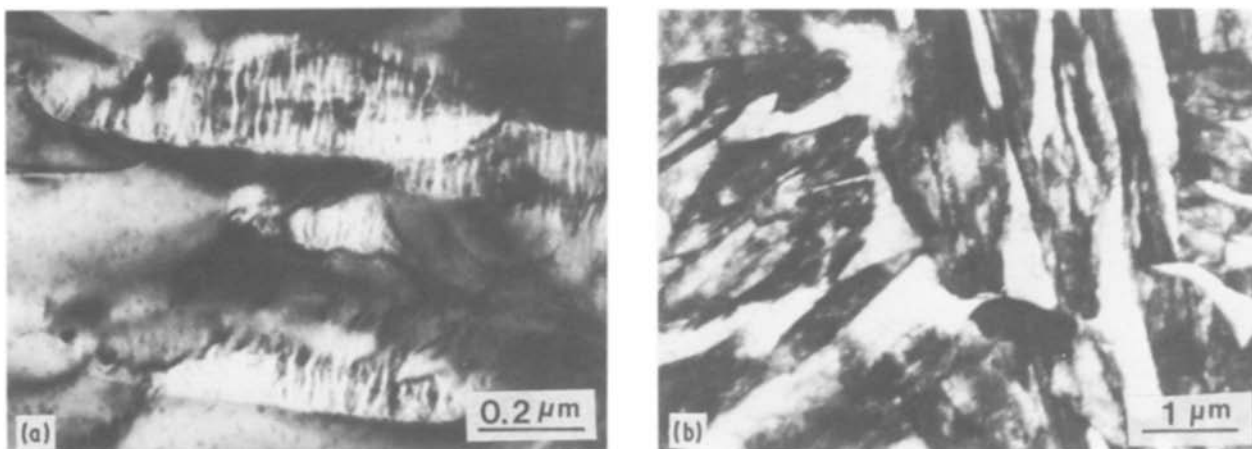


Figure 10 Transmission electron micrographs of the dual-phase steels: (a) Fe-0.15% C-3.01% Mn-0.51% Si-0.10% Mo, prepared by annealing for 10 min at 800°C, followed by water-quenching and tempering for 1 h at 700°C. (b) Fe-0.04% C-2.88% Mn, prepared by annealing for 10 min at 800°C and water-quenching.

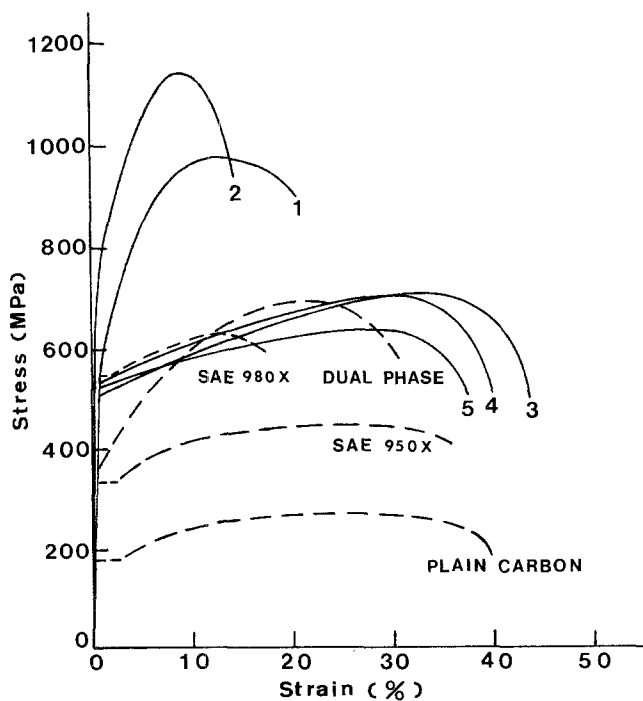


Figure 11 Some examples of the stress-strain curves of typical steels. (---) Plain-carbon, HSLA and dual-phase steels from [6] and [14]. (—) Results obtained in this work. Curve 1, Fe-0.13% C-2.88% Mn-0.56% Si-0.10% Mo steel, annealed 10 min at 700°C and air-cooled. Curve 2, Fe-0.13% C-2.88% Mn-0.56% Si-0.10% Mo steel, annealed 10 min at 750°C and air-cooled. Curve 3, Fe-0.15% C-3.01% Mn-0.51% Si-0.10% Mo steel, annealed 10 min at 800°C and water-quenched, then tempered 60 min at 700°C and air-cooled. Curve 4, Fe-0.08% C-3.07% Mn-0.23% Si steel, annealed 10 min at 800°C and water-quenched, then tempered 10 min at 700°C and air-cooled. Curve 5, Fe-0.11% C-2.89% Mn steel, annealed 10 min at 800°C and water-quenched, then tempered 30 min at 700°C and air-cooled.

elements containing steels prepared by water-quenching from 700 to 750°C: a lower mechanical strength and larger fracture elongation in comparison with those observed on air-cooled steels. This suggests that the precipitation hardening process by alloying elements has already begun during a simple air-cooling, while the alloying elements in the water-quenched steels are in a solid-solution state and they give low mechanical strength and high formability. In reality, we obtained $\sigma_R \sim 800$ MPa with $A_R \sim 30\%$ on molybdenum-bearing steels by water-quenching from 700°C, while $\sigma_R \sim 1000$ MPa was obtained with $A_R \sim 20\%$ on air cooling from the same temperature.

The effect of tempering on the mechanical properties of the dual-phase steels prepared by annealing for 10 min at 800°C followed by water-quenching, is seen as a remarkable softening occurring during the initial stage of heating from room temperature to 700°C. During the first several minutes of this process the martensite phase in ferrite grains is broken into fine particles but the equilibrium structure recovers after about 10 min, in which most of the fine martensite particles coagulate on grain boundaries and the mechanical strength is slightly restored.

The fracture elongation increases remarkably on tempering at 700°C and it attains a maximum value of about 40% after 10 min for the Fe-C-Mn and Fe-C-Mn-Si steels, while the mechanical strength remains almost constant at 700 MPa. In the case of steels containing an alloying element such as molybdenum, the precipitation occurs during tempering and fine precipitates are formed in the ductile ferrite matrix and thus the fracture elongation becomes more than 40% after 60 min tempering at 700°C with no loss of tensile strength (700 MPa). The work hardening coefficient, n , and the Vickers microhardness, H_v , change in a similar way to that of the mechanical strength. Thus, we obtained excellent mechanical properties: $\sigma_R \sim 700$ MPa with A_R more than 40% by tempering for 1 h at 700°C.

The austenite in dual-phase steels is a metastable phase and it transforms into the martensite by plastic deformation. We also confirmed this phenomenon by X-ray diffraction on cold rolled specimens. In general, this phase is identified by transmission electron microscopy as small particles, but in some cases as thin films between the martensite laths [5-11]. We observed that the volume fraction of the retained austenite becomes fairly large, near 10%, by intercritical annealing at low temperature (700°C) as shown in Figs 3, 4 and 6. These results can be interpreted, as already discussed by many workers, as the austenite formed at relatively low temperature being stabilized by the dissolved elements such as carbon, manganese and other alloying elements. Also, the work hardening coefficient (the uniform elongation) and the total elongation increase with increasing volume fraction of retained austenite. It appears consequently that the presence of the retained austenite is favourable to increasing uniform and fracture elongation by the mechanism of TRIP (transformation induced plasticity) [5, 12, 13].

In conclusion, our experimental results show that the addition of a small amount of refractory metal elements such as molybdenum, niobium and tantalum, which are used currently for HSLA steels, and boron is useful to improve the mechanical properties of high-manganese dual-phase steels developed in our previous work. This beneficial effect is primarily due to the microstructure in which the fine martensite phase is well dispersed in the ferrite matrix. The alloying elements further offer a large possibility to modify, by heat treatments under various conditions, the mechanical properties of these prepared steels.

Fig. 11 shows some typical examples of engineering stress-strain curves: the broken lines correspond to the alloys reported by Owen [6] and Morrow *et al.* [14], and the full lines represent some of our results in this work. Curves 1, 2 and 3 were obtained on the molybdenum-containing steels, nos 1 and 5, and curves 4, 5 on steels 7 (Fe-0.08% C-3.07%

Mn-0.23% Si) and 6 (Fe-0.11% C-2.89% Mn), respectively.

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