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Influence of deformation on the transformation of austenitic stainless steels

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Two commercial austenitic stainless steels of type 304 and 321 were deformed by reduction in thickness at room temperature or at liquid nitrogen temperature and subsequently annealed in the temperature range 473 to 1073 K. Microstructural changes were examined by electron microscopy. The deformation product in both the steels are different. An attempt has been made to correlate the mechanical properties with the microstructural changes.

1. Introduction

Martensite formation resulting from plastic deformation of metastable austenite is of great interest in producing high strength and ductility. Substantial strengthening can be obtained in metastable austenitic stainless steels by plastic deformation below M_d temperature (M_d is the $\gamma \rightarrow \alpha'$ transformation temperature induced by plastic deformation) to produce α' and ε martensite. The transformation to α' or ε depends upon alloy composition, stacking fault energy, degree of deformation etc. Stacking fault energy (SFE) of the austenite in Fe–Cr–Ni alloys depends upon the composition of the steel and generally ranges from 10 to 100 mJ min^{-2} [1]. Nickel and carbon tend to raise the SFE thereby influencing dislocation cross-slip while chromium, manganese, silicon and nickel tend to decrease the SFE of the austenite.

Mangonon and Thomas [2] reported that the amount of α' martensite in type 304 stainless steel increased with increasing strain at 77 K, reaching 50% at 20% strain. In addition, ε martensite was observed, increasing in amount up to 5% strain and then decreased with increasing strain. The volume fraction of α' increased steadily with increasing amount of deformation. Abrassart [3] and Remy and Pineau [4] investigated a 18 Cr, 7 Ni, 0.18 C stainless steel (in wt %) deformed at various temperatures. When the steel was 20% deformed at 423 K between $M_d(\gamma \rightarrow \alpha')$ and $E_d(\gamma \rightarrow \varepsilon)$, where E_d is the $\gamma \rightarrow \varepsilon$ transformation temperature induced by plastic deformation, twins and/or ε platelets begin to appear. After subsequent quenching to 203 K, α' martensite was observed. Coleman and West [5] rolled 16 Cr, 12 Ni stainless steel to 50% reduction in thickness at temperatures in the range of 423 to 195 K. The amount of α' martensite increased with decreasing deformation temperature, up to 85% at 195 K. Surprisingly, they did not report existence of ε martensite. Laptev and West [6] examined type 301, 302 and 316 stainless steel, rolled to 50% reduction in thickness in the temperature range 473 to 77 K. After 50% deformation at 77 K, the amount of α' martensite was 93% in 301, 76% in 302 and 28% in 316 steels. These authors did not report anything about ε martensite after deformation at 77 K in any of these steels.

The mechanism of hardening in austenitic stainless steels deformed at low temperatures and when they are subsequently heated (up to 673 K), is of considerable interest. Deformation of austenitic stainless steel under condition of intense cooling causes hardening due to the formation of α' phase, i.e. martensitic transformation. The volume fraction of the α' phase

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is considerably dependent upon the transformation temperature and amount of deformation or strain. Mangonon and Thomas [2] argued that strengthening depends not on the morphology of α' phase but upon the volume fraction of α' phase. Another possible mechanism of hardening in stainless steels could be associated with carbide precipitation. The precipitation of carbide could occur from the γ phase during the process of plastic deformation [7]. Another possible hardening mechanism is the "Suzuki effect", in which the solubility of foreign atoms is different in the stacking fault regions which have extended dislocations and the f c c matrix. The result is that the impurity atoms would diffuse into or out of the stacking fault regions, which require only a few atomic jumps. In either case the impurity atoms would lock the dislocations and thus would lead to hardening effect.

A detailed study of the reversion characteristics of martensite produced by refrigeration in an alloy of the same composition has been reported [4, 5, 8, 9]. Softening occurs by ageing at 773 K and above due to a decrease in per cent α', i.e. α' transforms back to γ phase. This transformation occurs by formation of subgrains or renucleation of γ within α' rather than by simple dissolution of α' phase [4, 5]. The material containing a large proportion of α' softens more rapidly than that containing less volume fraction of α' phase, therefore a greater degree of recrystallization is expected.

It is obvious that apart from the quenching temperature, the composition of the stainless steel plays an important role in the formation of α' or ε phases. There remains considerable uncertainty about the transformation sequence between the parent γ phase and the daughter phases during quenching at low temperatures and deformation of austenitic stainless steels. Thus, the purpose of the present investigation was to study the effect of deformation on the martensitic transformation in two different stainless steels. An attempt was also made to correlate the mechanical properties with development of microstructure during annealing.

2. Experimental procedure

Two commercial type 304 and 321 stainless steels were studied and their composition is given in Table I. These steels were solution treated at 1323 K for 1 h, water quenched and rolled at room temperature to 20, 40 and 60% reduction in thickness. Similarly, the 304 steel was rolled to 15, 30 and 50% reduction in thickness at low temperature by holding in liquid nitrogen for some time before giving a small amount of deformation. Specimens were annealed at 100 K intervals in the temperature range from 473 to 1173 K for 1 h, then quenched into water. Vickers hardness measurements were done with a 20 kg load. Tensile tests were performed in an Instron testing machine at a strain rate of approximately $1 \times 10^{-3}\,\mathrm{sec}^{-1}$ at room temperature and also at liquid nitrogen temperature.

Specimens for transmission electron microscopy were initially chemically thinned in a solution of 50% HCl, 10% HNO_3, 5% H_3PO_4 and 35% water. The final samples were prepared in a jet polisher with electrolyte of composition 40% acetic acid, 30% H_3PO_4, 20% HNO_3 and 10% water with 15 V at room temperature. X-ray diffractometer was employed to estimate the volume fraction of the second phases.

3. Results and discussion

3.1. Mechanical properties

During cold rolling, both steels exhibited rapid work hardening with hardness increasing from 174 to 240 VHN after 60% reduction (Figs. 1 and 2, respectively). Annealing the cold rolled steels for 1 h from room temperature to 673 K caused a slight increase in hardness. During annealing above 773 K, softening was observed. The samples rolled to 60% recrystallized rapidly in comparison with those rolled to 20% and 40%. As in Fig. 1, there was a significant increase in hardness of specimens rolled at 77 K, compared to those rolled at room temperature. On subsequent annealing, there was a sharp increase in hardness up to 673 K, followed by softening at higher temperature.

TABLE I Chemical composition (wt %) of steels investigated

Type	% Cr	% Ni	% Mn	% Ti	% Si	% V	% C	% Fe
304	18.8	11.5	–	–	< 1	–	0.07	Balance
321	18.2	9.0	1.3	0.16	< 1	0.05	0.019	Balance

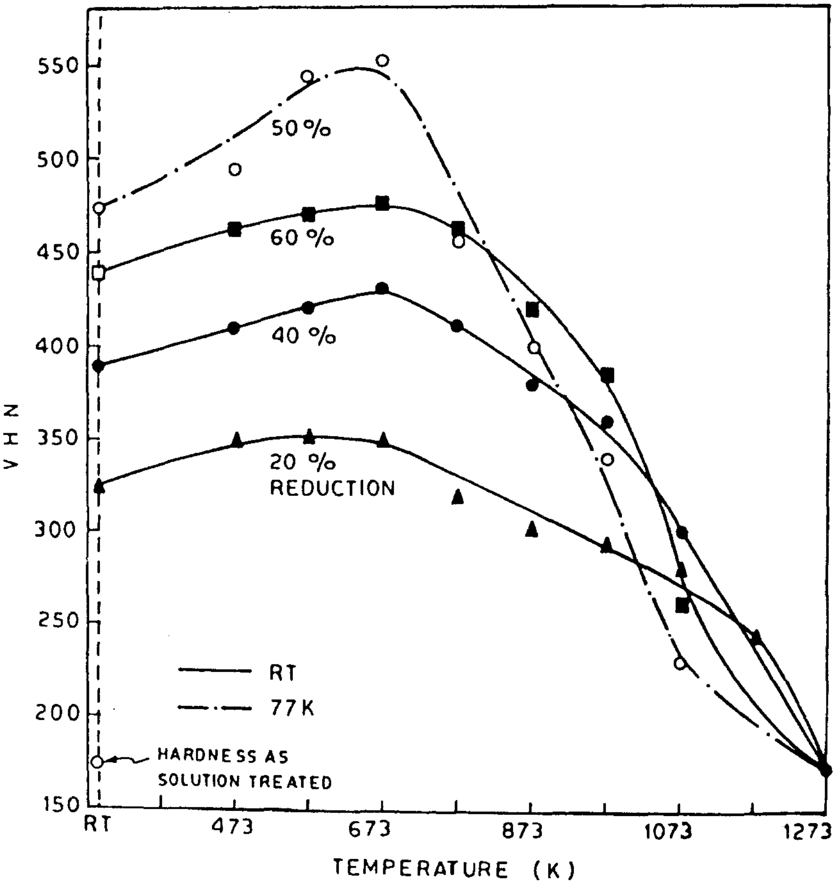

Figure 1 Isochronal hardness curves of deformed 304 type austenitic stainless steel.

Tensile properties of steels are given in Tables II to IV. There was very little increase in yield strength and ultimate tensile strength (UTS) up to 673 K and thereafter strength decreased with increasing temperature. Tensile properties are consistent with the changes in hardness as seen in Figs. 1 and 2.

It can be readily seen that both 304 and 321 steels have similar changes in hardness on cold working at room temperature as well as on annealing, but their deformation process is different. On the basis of microstructure, the former steel gives α' martensite and the latter gives only deformation bands or twin bands. The fact that the α' does not increase on anneal ing the 304 steel, shows that α' is not responsible for anneal hardening.

At higher ageing temperatures above 673 K,

TABLE II Tensile properties of type 304 stainless steel after deformation at room temperature and subsequent annealing

Annealing temperature (K)	YS (MPa)		UTS (MPa)		% elongation	
	40%	60%	40%	60%	40%	60%
As-deformed	999	1120	1117	1169	9	8
473	1044	1020	1128	1207	13	6
573	1054	1083	1148	1197	11	7
673	1044	1108	1133	1208	11	6
773	926	1025	1103	1162	22	7
873	857	827	1025	985	22	12
973	837	798	1015	1005	27	24
1073	414	489	759	995	51	29

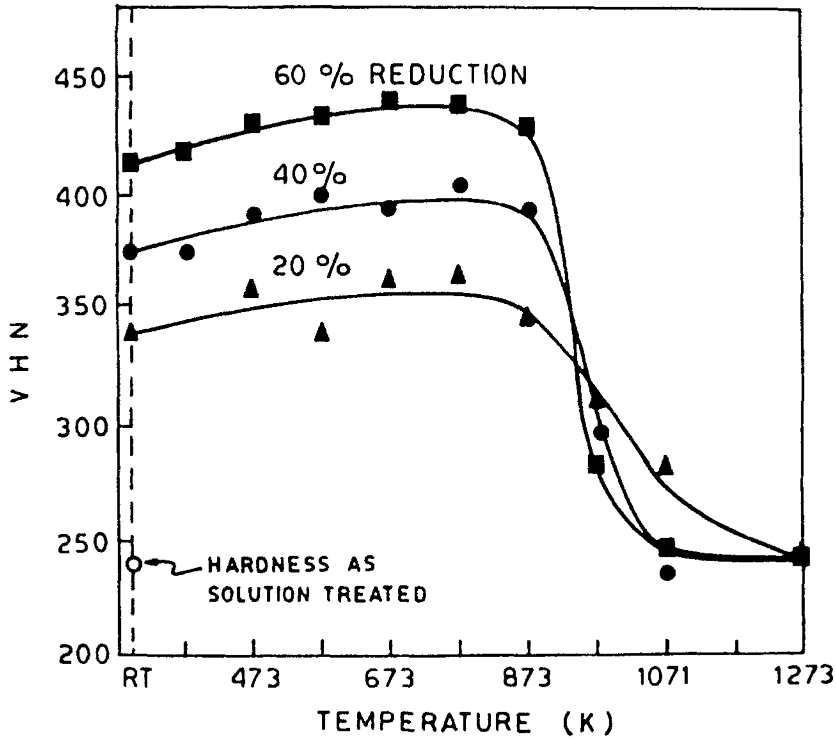

Figure 2 Isochronal hardness curves of deformed 321 type austenitic stainless steel.

there was a stress relief effect on the mechanical properties. Stress relief ageing was due to reversion of α' phase into γ phase.

3.2. Microstructural observation

An electron micrograph from the 304 steel specimen strained 4% at room temperature shows closely spaced stacking faults (Fig. 3a). The microstructure of the specimen strained 4% at 77 K is shown in Fig. 3b. These faults appeared to be localized h c p (ε) regions containing packets of α' martensite.

The microstructure of 304 steel specimens rolled 60% at room temperature, showing a high density of dislocations and packets of α' phase is seen in Fig. 4. Traces of ε phase were not found at this high level of deformation by TEM as well as X-ray diffraction analysis. The volume fraction of α' phase was found by X-ray diffraction analysis to be 20%. As pointed out by Mangonon and Thomas [2], the volume fraction of ε phase increased up to 5% strained at 77 K, and then decreased with further increasing strain. In the present case it is quite possible that under heavy deformation, any ε phase if formed, might have transformed to α' phase. The existence of α' phase was observed up to an annealing temperature of 673 K, as shown in Fig. 5. Specimens rolled 60% at room temperature were not fully recrystallized after annealing at 1073 K. Annealing twins were observed at 1073 K as shown in Fig. 6.

In type 304 steel rolled 50% at 77 K, TEM analysis shows the presence of both ε and α'

TABLE III Tensile properties of type 304 stainless steel after deformation at 77 K and subsequent annealing

Annealing temperature (K)	YS (MPa)		UTS (MPa)		% elongation (mm)	
	30%	50%	30%	50%	30%	50%
As-deformed	1399	1374	1487	1453	5	7
473	–	1468	–	1527	–	6
673	1300	1635	1359	1686	5	6
873	847	768	1113	1143	12	8

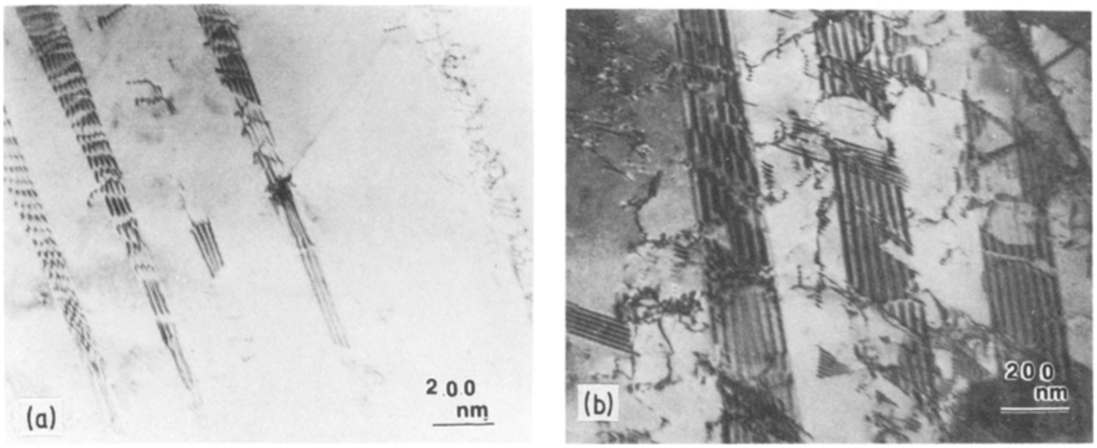

Figure 3 Electron micrographs of tensile sample strained 4% at (a) room temperature showing stacking faults, and (b) 77 K showing closely spaced stacking faults.

martensites. The ε martensite could not be observed by X-ray diffraction analysis as the volume fraction was very small. The volume fraction of α' phase was large compared to that in the specimens deformed at room temperature and is approximately 63% after 50% deformation at 77 K. The ε martensite persisted up to an annealing temperature of 473 K (Fig. 7). Fig. 7b is a dark-field micrograph from one of the hcp $(0\bar{1}\bar{1}\bar{1})_\varepsilon$ reflection, showing the presence of ε martensite phase. Mangonon and Thomas [2] also reported the existence of ε martensite up to 473 K. In addition they claimed that during annealing up to 473 K, ε phase transformed to α' phase, thus increasing the amount

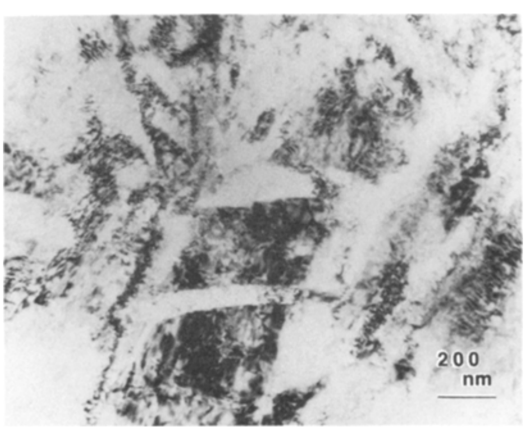

Figure 4 Electron micrographs of specimens of 304 type austenitic stainless steel rolled 60% at room temperature showing high density of dislocations and packets of α' martensite.

of α' phase. In contrast, in the present investigation the amount of α' phase was decreased from 63% in the as-deformed sample at 77 K, to 53% after annealing at 473 K. On annealing up to 673 K, only α' phase was observed and the volume fraction of α' phase was further reduced to 43%. The microstructure shown in Fig. 8 corresponds to the peak hardness. Specimens deformed at 77 K were not fully recrystallized by annealing at 1073 K. Low-angle boundaries and annealing twins were observed after this treatment, as shown in Fig. 9.

Fig. 10 is an electron micrograph from 321 steel strained 10% at 77 K, showing the presence of only twins. The presence of α' or ε martensite was not observed.

The mictrostructure of 321 steel rolled 60% at room temperature, shows a high density of dislocations and elongated bands (Fig. 11). These bands could be deformation twins. The maximum thickness of the bands was about 300 nm. The presence of α' or ε martensite was not observed by TEM and X-ray diffraction. On subsequent annealing at 673 K, twins become more distinct (Fig. 12). This microstructure corresponds to peak hardness. After annealing to 1073 K for 1 h, twins and dislocations are still present in the unrecrystallized region (Fig. 13).

In austenite, twins and ε martensite can be formed, respectively, by the superposition of stacking faults either on every (1 1 1) plane or every second (1 1 1) plane. Deformation twins or thin ε platelets show very similar morphologies, therefore, structure identification must be

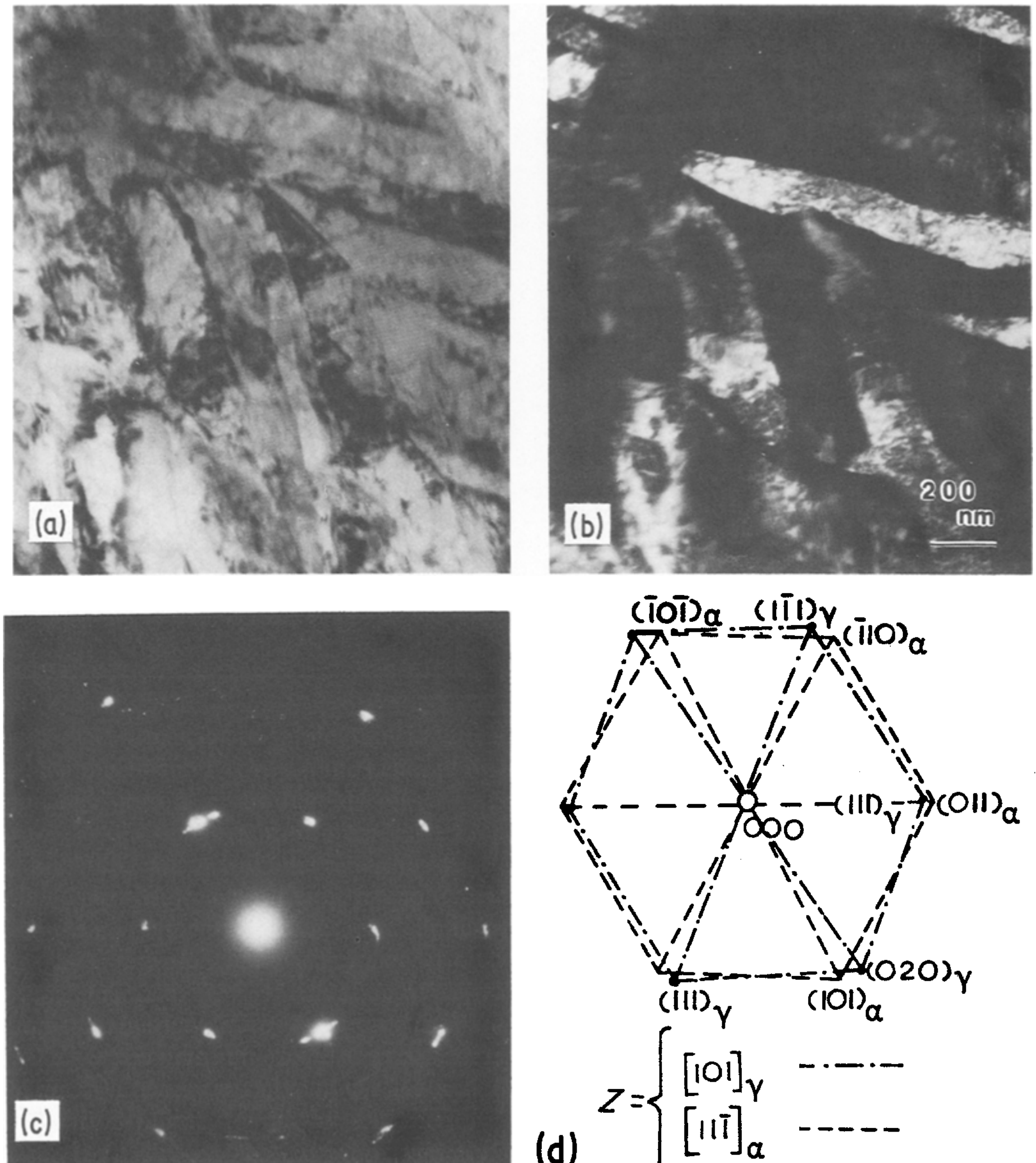

Figure 5 Electron micrographs of specimens of 304 type austenitic stainless steel rolled 60% at room temperature and annealed at 673 K for 1 h: (a) bright-field, showing α' phase and high density of dislocations in the matrix; (b) dark-field, from $(\bar{1}\,1\,0)_\alpha$ reflection; (c) diffraction pattern; (d) key.

TABLE IV Tensile properties of type 321 stainless steel after deformation at room temperature and subsequent annealing

Annealing temperature (K)	YS (MPa)		UTS (MPa)		% elongation	
	40%	60%	40%	60%	40%	60%
As-deformed	1005	1059	1113	1259	6	6
473	993	968	1129	1239	6	7
573	1003	1054	1084	1241	6	6
673	933	1074	1153	1314	7	5
773	973	999	1107	1281	7	6
873	784	837	959	1027	13	9
973	744	705	963	975	16	2
1073	546	621	977	936	30	32

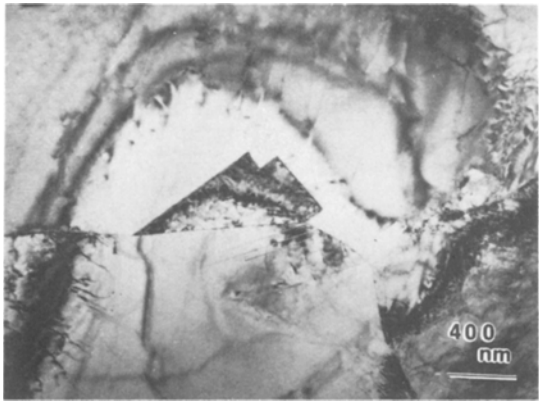

Figure 6 Electron micrograph of specimens of 304 type stainless steel rolled 60% at room temperature and annealed at 1073 K for 1 h showing the specimen is not fully recrystallized.

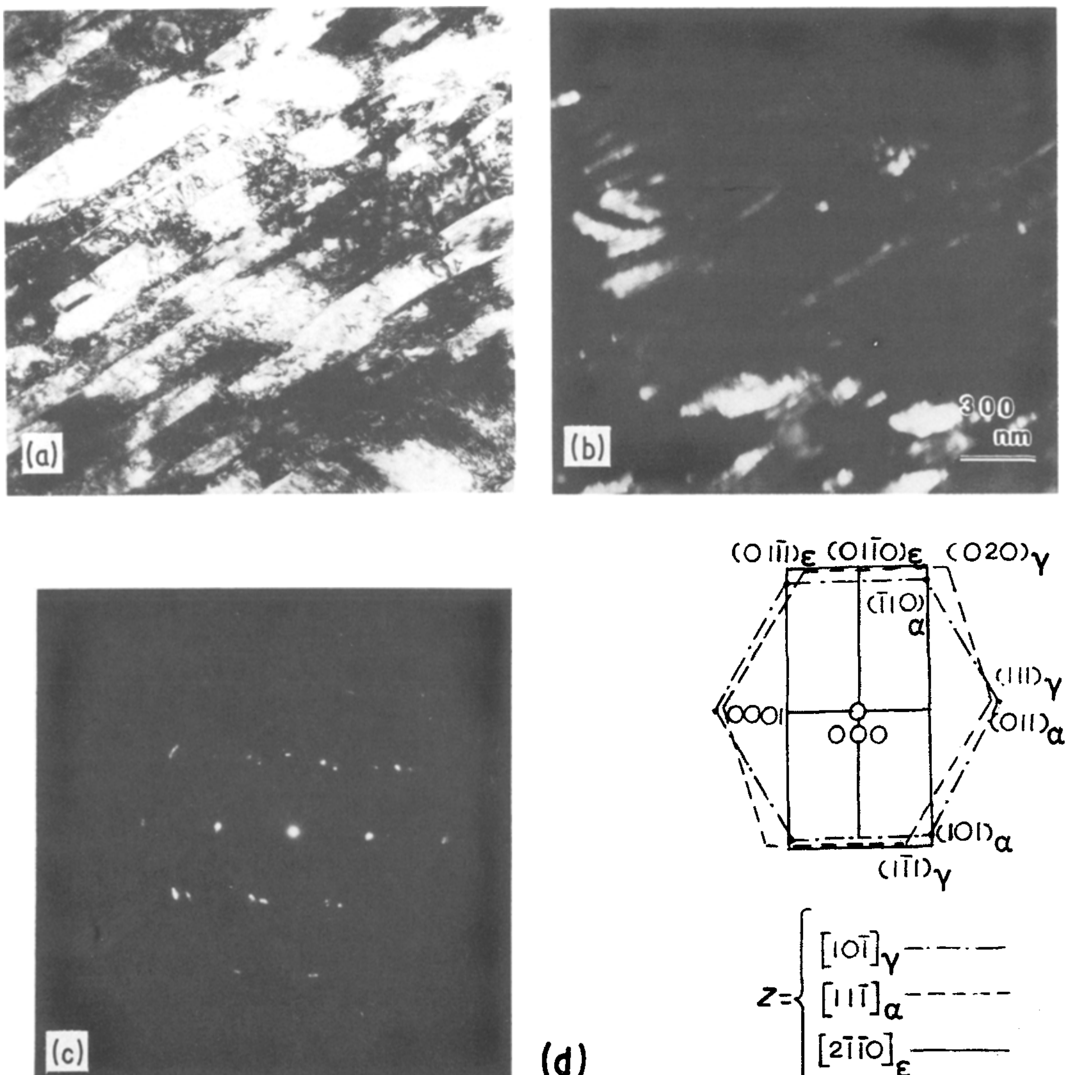

Figure 7 Electron micrograph of specimens of 304 type stainless steel rolled 60% at 77 K and annealed at 473 K for 1 h showing the coexistence of α' and ε phase in the matrix: (a) bright-field; (b) dark-field from $(0\,1\,\bar{1}\,1)_\varepsilon$ reflection showing the ε phase; (c) diffraction pattern; (d) key.

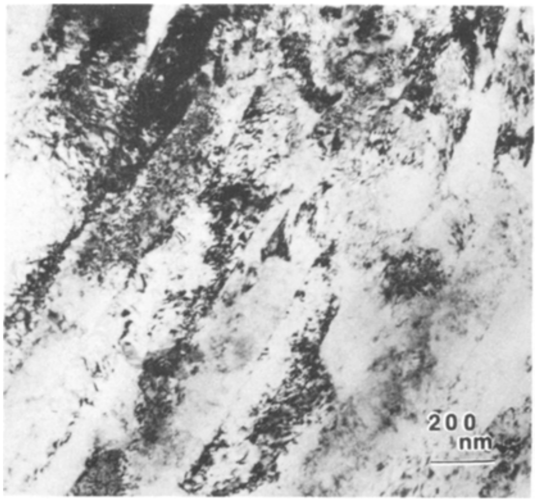

Figure 8 Electron micrograph of the specimen of 304 type stainless steel rolled 60% at 77 K and annealed at 673 K for 1 h showing high density of dislocations and packet of α' martensite.

carried out by systematic diffraction techniques [10]. A number of reasonably low-order matrix orientations were used and confirmed the above results. In addition, Mangonon and Thomas [2] reported that ε platelets are stable only up to 473 K. The present results show the presence of twins up to 1073 K which further confirms the above findings.

The formation of twins during severe cold working can be attributed to both relatively low stacking fault energy and solid solution

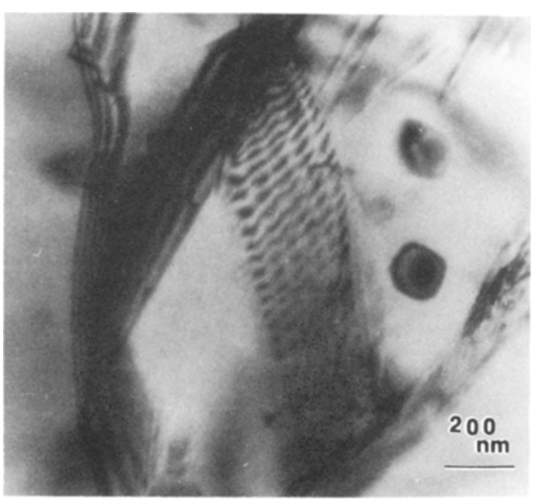

Figure 9 Electron micrograph of the specimens of 304 type stainless steel rolled 60% at 77 K and annealed at 1073 K for 1 h showing the specimen is not fully crystallized.

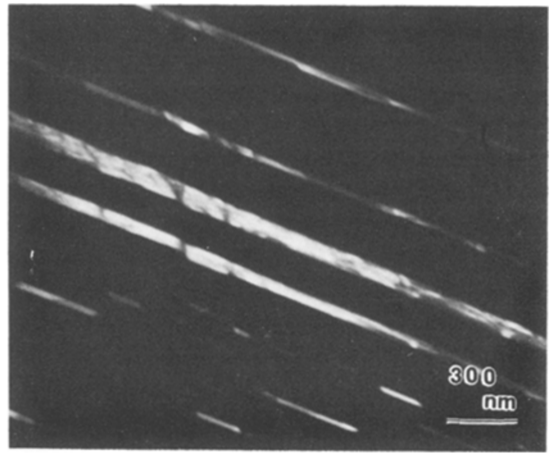

Figure 10 Dark-field electron micrographs of tensile sample of 321 type stainless steel strained 10% at 77 K from one of the twin reflection showing the presence of twins.

strengthening caused by the variety of substitutional elements in the alloy [11–13]. It has been demonstrated that the alloying elements which reduce the stacking fault energy, favour the nucleation of twins at lower stresses. It is quite possible that the presence of titanium also further lowers the stacking fault energy in the 321 stainless steel, compared to 304 stainless steel and thus produces the deformation twins.

On comparing the microstructure of both steels, it is interesting to note that 304 steel shows the $\gamma \rightarrow \alpha'$ transformation in the specimens rolled to 60% at room temperature, while 321 steel shows the transformation of fcc to twins. On deforming both the steels at 77 K, only 304 steel shows the $\gamma \rightarrow \alpha'$ and $\gamma \rightarrow \varepsilon$ transformation. Even after small strain (5%), 304 steel shows both α' and ε phases, whereas 321 steel did not show the presence of either α' or ε phase, even after subjecting to further strain (10%) at 77 K. On giving significant amount of deformation (50%) at low temperature 304 steel shows a large volume fraction of α' phase as compared to room temperature deformation, thereby increasing the hardness and strength values. On subsequent annealing up to 673 K there was no further increase in volume fraction of the α' phase in 304 steel. It further suggests that the initial transformation sequence was $\gamma \rightarrow \varepsilon \rightarrow \alpha'$, while on annealing, the sequence of transformation appeared to be $\varepsilon \rightarrow \gamma$ and $\alpha' \sigma \gamma$. X-ray analysis did not show any increase in the volume fraction of the α' phase, rather a

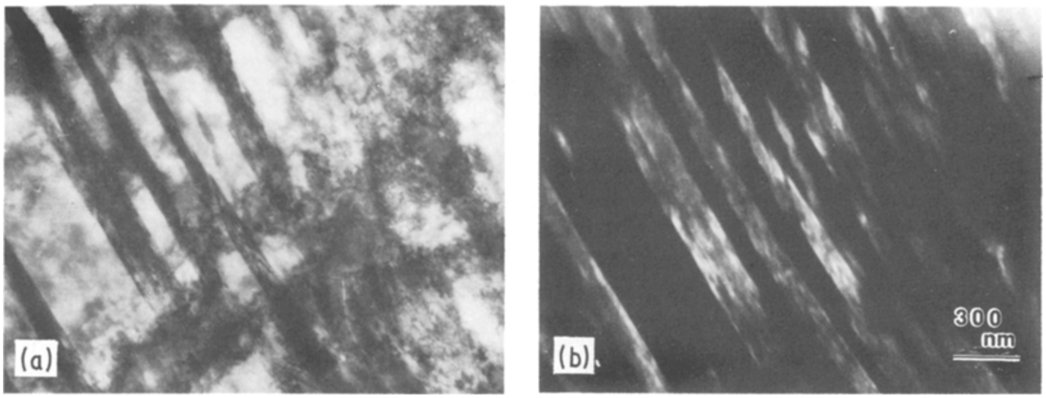

Figure 11 Electron micrographs of specimens of 321 type stainless steel rolled 60% at room temperature showing a high density of dislocations and deformaton bands: (a) bright-field; and (b) dark-field.

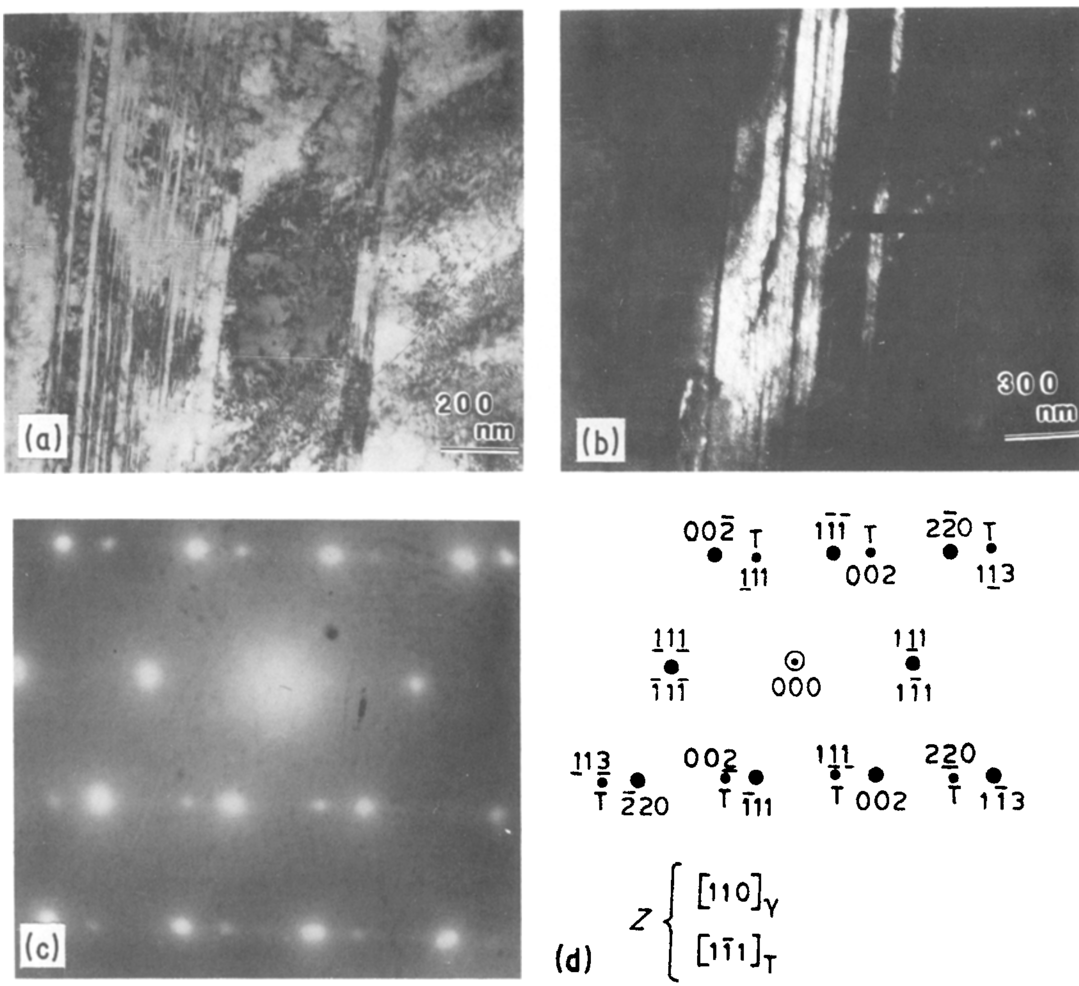

Figure 12 Electron micrographs of specimens of 321 type stainless steel rolled 60% at room temperature and annealed at 673 K for 1 h showing the presence of twins: (a) bright-field; (b) dark-field from one twin reflection; (c) and (d) are the corresponding diffraction pattern and indexing.

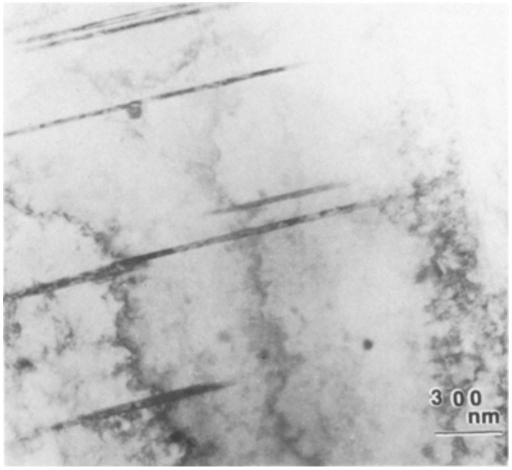

Figure 13 Electron micrograph of specimens of 321 type stainless steel rolled 60% at room temperature and annealed aty 1073 K for 1 h showing the presence of twins.

decrease in the volume fraction of α' phase from 63% to 53% after annealing at 473 K and a further reduction to 43% at an annealing temperature of 673 K. The basic composition of both steels are the same except for the addition of titanium in 321 steel. The 321 steel did not show the presence of any α' or ε martensite on deforming at room temperature or 77 K. It suggests that small differences in composition can change the mechanism of deformation.

4. Summary and conclusions

The hardness of 304 stainless steel deformed 50% at 77 K was greater than that at room temperature deformation. This was due to increased dislocation density, α' phase and ε' phase at the low temperature deformation. The hardness curves for both stainless steels are comparable, but hardening products in both the steels are different. The hardening in the austenitic stainless steels is achieved not only by martensite production in the fcc phase but also by deformation bands. These deformation bands are more stable and can survive annealing up to 1073 K in the unrecrystallized region. The ε' phase is stable up to 473 K and the α' phase is stable up to 673 K.

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