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Effect of prior deformation on Co–Ti, Co–Ti–Nb and Co–Ti–Fe alloys

JOGENDER SINGH
Department of Mechanical Engineering, University of Illinois at Urbana-Champaign, 1206 West Green Street, Urbana, Illinois 61801, USA

The influence of prior deformation in spinodally decomposing Co–Ti, Co–Ti–Nb and Co–Ti–Fe alloys has been investigated using transmission electron microscopy and hardness isotherms. Deformation seems to have an influence on the decomposition sequence in Co–3% Ti alloy; however, it does not affect the sequence of transformations in the ternary alloys. The ternary alloys show peak hardness after deformation and subsequent annealing at 873 K. The peak hardness of the deformed alloys occurred quite early, compared with the undeformed alloys.

1. Introduction

Spinodal transformation has attracted considerable attention in recent years. Besides the intrinsic scientific interest offered by the nuances of this phase transformation, it also has technological potential for the design of strong alloys. Ditchek and Schwartz [1] have reviewed these aspects in a succinct fashion. The spinodal decomposition in binary Co–Ti and ternary Co–Ti–Fe and Co–Ti–Nb alloys has been well established [2–7]. The effect of minor additions of lanthanum, niobium and iron has been elucidated [6–8]. Long-term ageing, leading to a discontinuous transformation, has been explored [9, 10]. In this communication the influence of prior deformation on spinodal transformation in these alloys is presented and discussed.

It is pertinent to point out that there have been few studies of the effect of prior deformation on spinodal decomposition. An early paper by Plewes [11] emphasized the advantages of prior deformation in the Cu–Ni–Sn system in achieving excellent combination of yield limit and fracture ductility. Dutkiewicz [12, 13] has reported that prior deformation causes a significantly greater strengthening in Cu–Ti alloys and the spinodal curve is shifted to lower temperatures. It also increases the rate of discontinuous precipitation. Spooner and Lefevre [14] studied Cu–15 Ni–8 Sn alloy and concluded that deformation prior to ageing produces no major structural or kinetic change in the spinodal decomposition and coarsening. However, it led to accelerated strengthening.

2. Experimental procedure

The experimental alloys were supplied by Cobalt Information Centre, Belgium in the form of rods 15 cm long and 2 cm diameter. Their compositions are Co–3 wt % Ti, Co–3 wt % Ti–1 wt % Nb, Co–5 wt % Ti–2 wt % Nb, Co–3 wt % Ti–1 wt % Fe and Co–3 wt % Ti–2 wt % Fe. In order to have materials of convenient dimensions for subsequent mechanical testing and microstructural observation, the alloys were hot forged followed by cold rolling with intermediate heating at 1073 K and quenching.

All the samples were encapsulated in silica tubes under vacuum and solutionized at 1473 K for 1.5 h and quenched in water. To study the effect of deformation on the ageing behaviour of these alloys, the samples were deformed by cold rolling at room temperature to 40% reduction in thickness after solutionizing and water quenching and subsequent to rolling aged at 873 and 973 K for different times.

Thin foils for electron microscopic examination were prepared by utilizing a Fischione twin-jet polisher. A solution of 50% orthophosphoric acid in distilled water was used for electropolishing. Foils were examined in a Philips EM-300 electron microscope operating at 100 kV.

3. Results and discussion

3.1. Mechanical properties

The variation of hardness with ageing at 873 K for Co–3 wt % Ti alloy deformed by rolling to 40% reduction in thickness is shown in Fig. 1. An increase in hardness value due to deformation over the value for the as-quenched state was observed. The increase in hardness was related to the presence of a high density of dislocations introduced by deformation. On ageing at 873 K up to 12 h, there was no change in the hardness values of the deformed sample. In addition, after ageing at 873 K, the incremental hardness of the deformed sample was less than that of the undeformed alloy.

The variation of hardness with ageing at 873 K for Co–3% Ti–1% Nb and Co–3% Ti–2% Nb alloys is shown in Fig. 2. An increase in hardness values due to deformation over the values for the as-quenched state was observed. This effect was greatest for the Co–3% Ti–2% Nb alloy. On ageing at 873 K, the hardness varies in the same way for the alloys with 1% and 2% Nb. The increase in hardness up to peak hardness for the deformed alloys was comparable with that observed without deformation as shown in Table I. However, the absolute peak hardness values for both

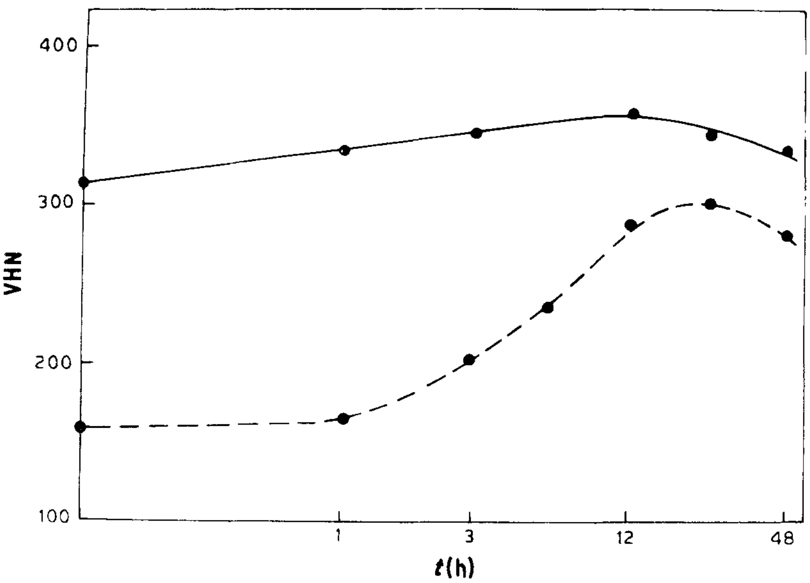


Figure 1 Isothermal hardness plots for Co–3% Ti alloy on ageing at 873 K with and without 40% prior deformation. --- Undeformed, —— deformed.

the deformed alloys were substantially higher as compared to those for the undeformed alloys [8]. Microstructural evolution suggested that the peak hardness was due to composite effects of a high density of dislocations and precipitation. Finally, as expected, the peak in hardness at 873 K was reached in 3 h for the deformed alloys where as only a continuous increase was observed for the undeformed alloys for ageing up to 48 h [8].

The results of the investigation on the Co–3% Ti–1% Fe alloys and Co–3% Ti–2% Fe alloys are presented in Fig. 3. The increase in hardness values due to deformation over the values for the as-quenched samples [6] show a similar behaviour and can be explained on the same basis as in the case of the Co–Ti–Nb alloys. The ageing behaviour of the Co–Ti–Fe system was quantitatively very similar to that of the Co–Ti–Nb systems at 873 K.

3.2. Microstructural changes

The effect of prior deformation on the microstructural evolution during the ageing of the binary as well as ternary alloys at 873 K was studied. Fig. 4a illustrates the presence of dislocations and the formation of a substructure in the as-quenched and deformed binary

TABLE I Increase in hardness on ageing of deformed and undeformed alloys at 873 K

Alloys	Increase in hardness (VHN)	
	Deformed	Undeformed
Co–3% Ti	45	143
Co–3% Ti–1% Nb	118	153
Co–3 Ti–2% Nb	155	155
Co–3% Ti–1% Fe	82	96
Co–3% Ti–2% Fe	56	98

alloy. On ageing at 873 K for 30 min, non-uniform precipitation was observed along with the presence of dislocations (Fig. 4b). The microstructure does not reveal the occurrence of modulations. On continued ageing for 3 h, the deformed sample recrystallized partially with a relatively large amount of precipitation in the recrystallized region (Fig. 4c).

Fig. 5a shows the microstructure of the Co–3% Ti–2% Nb alloy deformed to 40% reduction in thickness by rolling, showing the development of elongated bands and a high density of dislocations. On subsequent ageing at 873 K for 30 min, modulations with a poor contrast were apparent within the bands along with a high density of dislocations (Fig. 5b). When

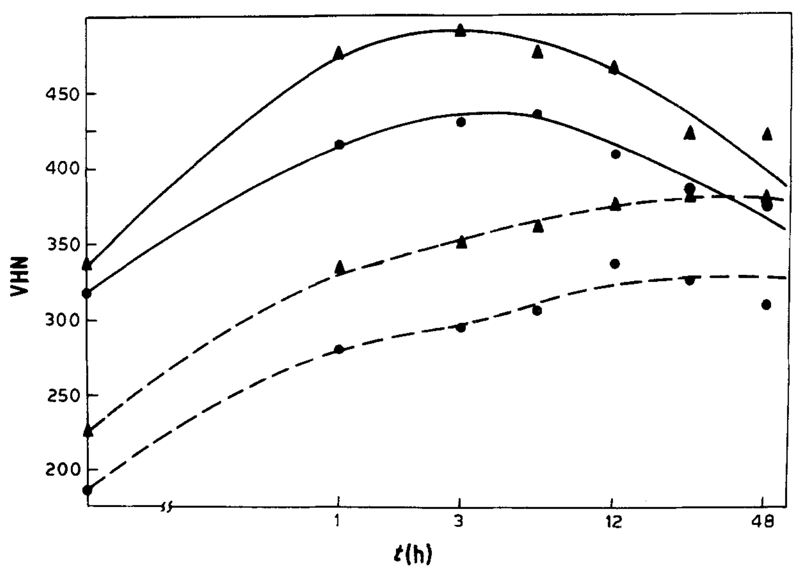


Figure 2 Isothermal hardness plots for ● Co–3% Ti–1% Nb and ▲ Co–3% Ti–2% Nb alloys on ageing at 873 K with and without 40% prior deformation. --- Undeformed, —— deformed.

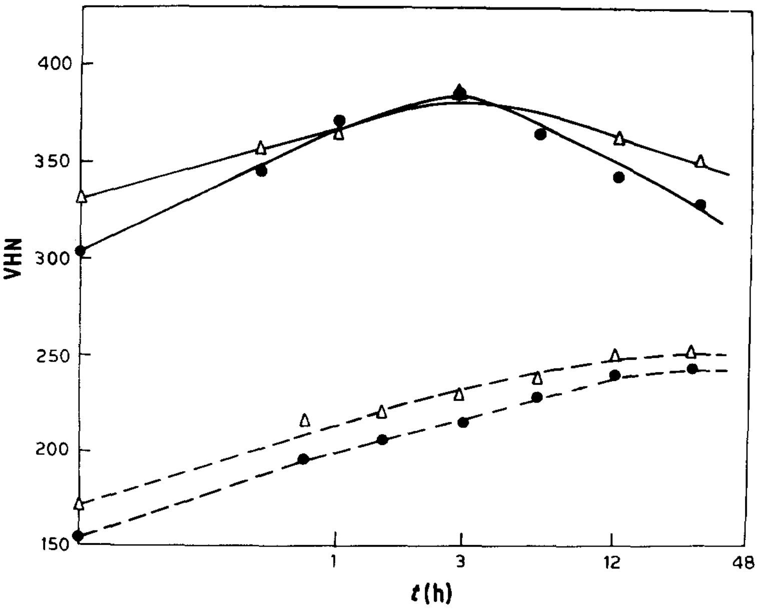


Figure 3 Isothermal hardness plots for ● Co–3% Ti–1% Fe and ▲ Co–3% Ti–2% on ageing at 873 and 973 K with and without 40% prior deformation. --- Undeformed, —— deformed.

ageing was continued up to 3 h, the resulting microstructure showed cuboidal precipitates (Fig. 5c). The deformation bands were seen to persist.

The microstructure of the Co–3% Ti–2% Fe after 40% deformation is comparable to that of Co–3% Ti and Co–3% Ti–2% Nb. After ageing for 30 min at 873 K, modulations were seen (Fig. 6a). Ageing for 3 h led to the formation of cuboidal particles (Fig. 6b) and partial recrystallization (Fig. 6c). After 24 h, a completely recrystallized structure with a heterogeneous distribution of coarse globular particles was seen (Fig. 6d).

The electron micrographs of the deformed binary as well as ternary alloys show a rather similar development of elongated bands and a high density of dislocations (Figs. 4a and 5a). On ageing at 873 K for 30 min, the supersaturated solid solution in the case of ternary alloys decomposed into a two-phase mixture exhibiting modulations. The contrast of the modulations was poor due to the residual dislocation bands (Figs. 5b and 6a). However, under similar ageing conditions, the binary alloy did not reveal the presence of modulations. Instead the occurrence of heterogeneous precipitation was observed along with a high density of dislocations (Fig. 4b). Dutkiewicz [12, 13] has suggested that deformation has no effect on spinodal decomposition but does have an influence on other types of transformation such as discontinuous precipitation and recrystallization. Furthermore, in conformity with his observations, the results on the ternary alloys show that decomposition was accelerated because of enhanced diffusion due to the increase of vacancy concentration arising due to deformation. In contrast, deformation does seem to have an influence

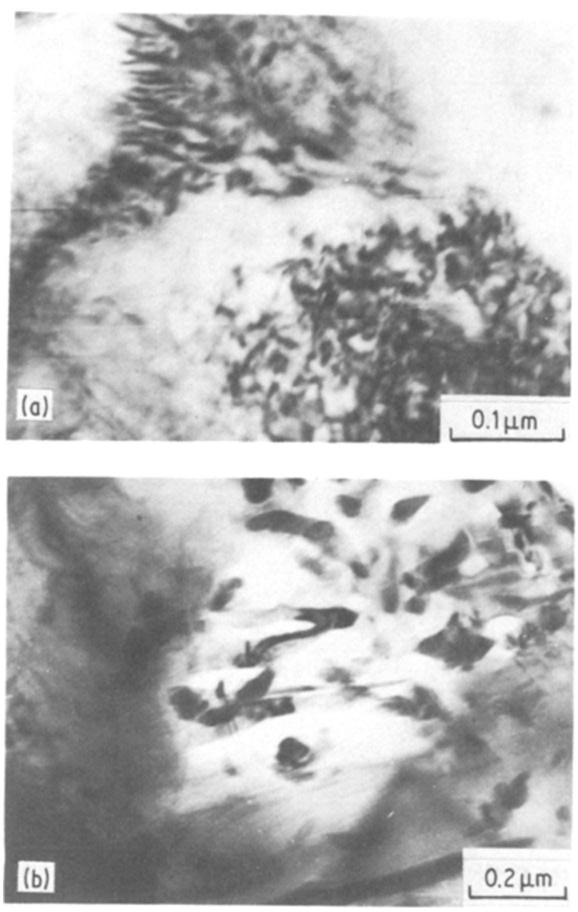
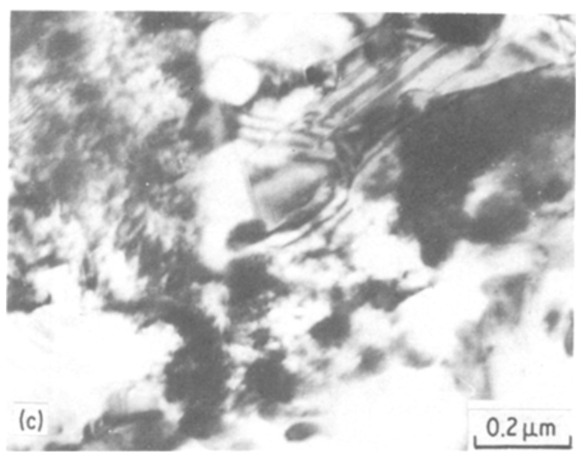
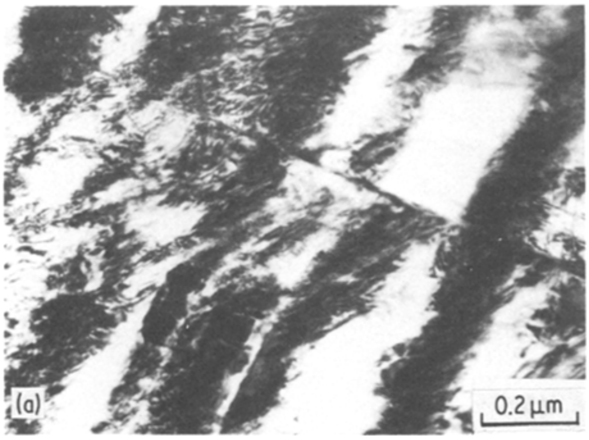
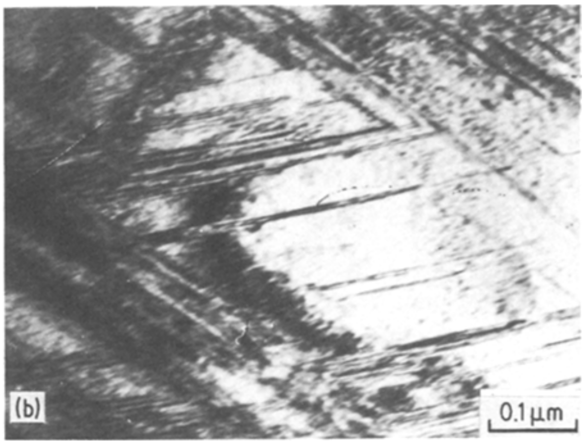


Figure 4 Transmission electron micrographs of Co–3% Ti alloy deformed 40% and subsequently aged at 873 K. (a) As-deformed: showing high density of dislocations and their distribution. (b) After 0.5 h: showing heterogeneous precipitation in the recrystallized region and dislocations. (c) After 3 h: showing increased precipitation in the recrystallized region and un-recrystallized region with dislocations.







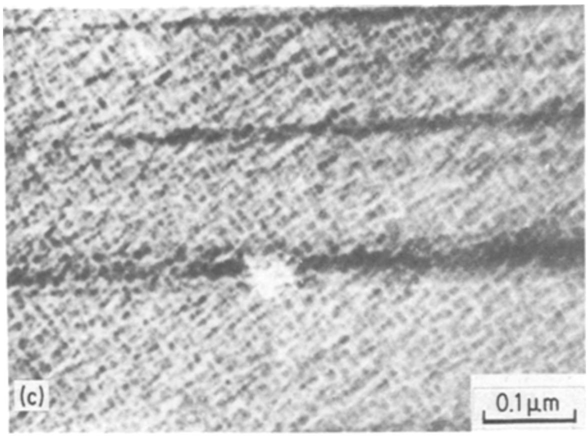


Figure 5 Transmission electron micrographs of Co–3% Ti–2% Nb alloy deformed 40% and aged at 873 K. (a) As-deformed: showing high density of dislocations and deformation bands. (b) After 0.5 h: showing modulations and deformation bands. (c) After 3 h: cuboidal precipitates and deformation bands.

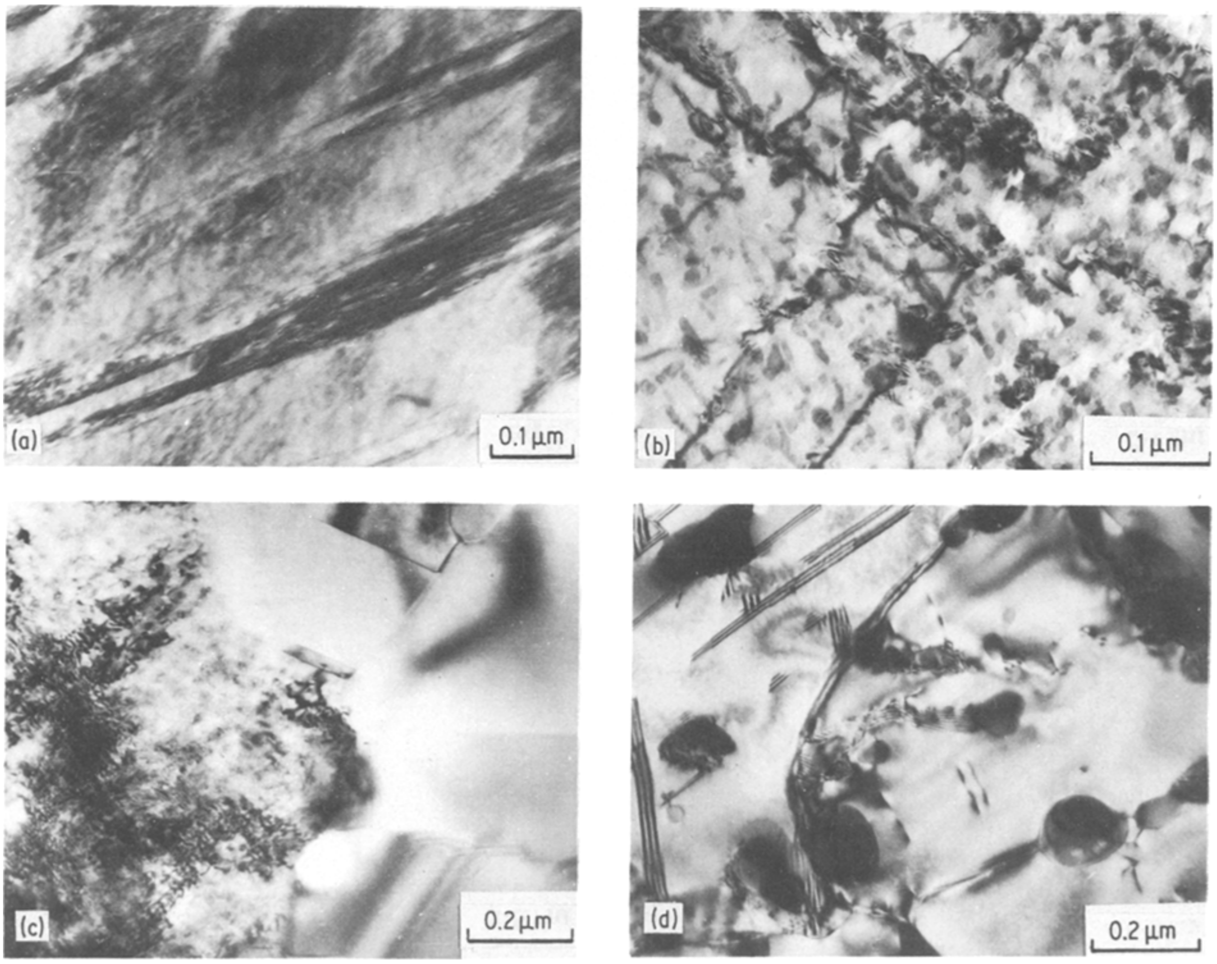


Figure 6 Transmission electron micrographs of Co–3% Ti–2% Fe alloy deformed 40% and aged at 873 K. (a) After 0.5 h: showing modulations with poor contrast and deformation bands. (b) After 3 h: cuboidal precipitates with dislocations. (c) After 3 h: partial recrystallization of the deformed alloy. (d) After 24 h: showing heterogeneous precipitation and recrystallized matrix with stacking faults.

on the decomposition tendency of the Co–3% Ti binary alloy. This difference is related to the binary alloy having a composition near the spinodal and thus being subject to changes in the sequence of transformation on ageing at 873 K after deformation. However, deformation does not have any influence on the sequence of transformation in the ternary alloys having compositions well within the spinodal. On continued ageing for 3 h, the resulting microstructure of the ternary alloy at the peak hardness values shows cuboidal precipitates along with deformation bands (Figs. 5c and 6b). It is interesting to note that even after deformation and ageing, the peak hardness of the ternary alloys occurred during the third stage of coarsening, although this stage of coarsening occurred much earlier as compared to the undeformed and aged alloys. With longer ageing, recrystallization was observed to take place in the binary alloy as also Co–3% Ti–2% Fe alloy. Precipitates with a coarse globular morphology were distributed within the recrystallized matrix. Invariably stacking faults were attached to the precipitates. A similar behaviour is also expected for the Co–3% Ti–2% Nb alloy.

Owing to the poor contract of the modulations for the deformed alloys, it was difficult to measure the wavelength of the modulations in these alloys and hence it was not possible to find the rate of reaction. However, from the fact that the peak hardness as well as the third stage of coarsening are reached earlier for the deformed alloys, compared to the undeformed alloys [7], it can be concluded that the kinetics of the decomposition reaction in deformed alloys was faster in comparison with that in the undeformed alloys.

4. Conclusions

Deformation prior to ageing influences the sequence of transformation in the dilute binary Co–3% Ti alloy, while it does not have any effect on the transformation sequence in the ternary Co–3% Ti–2% Nb and Co–3% Ti–2% Fe alloys. The kinetics of transformation is, however, accelerated due to defects introduced by the deformation process.

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