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Plastic deformation of CuAlMn shape-memory alloys

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CuAlMn alloys transforming below room temperature were cold rolled after quenching up to 93% reduction. Alloys deformed only 10% were already completely martensitic and did not show reverse transformation to β_1 upon heating. With increasing deformation, refinement of martensitic plates occurred due to repeated internal twinning and injection of new orientation variants. This was accompanied by decreasing size of ordered domains down to that of short-range ordered microdomains at 93% reduction. Deformation bands appearing at 60% reduction consisted of extremely small grains, giving an electron diffraction pattern resembling that of an amorphous structure.

1. Introduction

There is a considerable interest in plastic deformation of shape-memory alloys [1–8], although it is not a desired effect in shape-memory applications. This problem is important, however, from the point of view of degradation of reversible martensitic transformation, the role of ordering, interaction of grain boundaries and evolution of deformation bands.

In most works, the effect of plastic deformation was studied in CuZnAl alloys [1–5] in the deformation range up to about 40%. Within this transformation range, reverse martensitic transformation was observed, however, showing much enlarged transformation hysteresis [2, 4, 5]. Complete degradation of the transformation occurred at about 30% reduction by rolling [4, 5]. This phenomenon was also called, by Zhang and Hornbogen [2], stabilization of martensite, because heating of a deformed sample does not induce reverse transformation. The following deformation morphologies were reported [1, 2]: variant–variant coalescence, stress-induced martensite to martensite transformation, injection of foreign variants to plate groups, internal twinning and slip. The deformation occurred mainly by shear on the $(001)_0$ basal plane in the $[010]_0$ direction [3].

Another factor influencing martensitic transformation temperatures is the changing degree of order due to passing of slip dislocations, and changing atomic arrangement [2, 3].

A detailed study of the effect of a grain boundary on pseudo elastic deformation of CuZnSi bicrystals [6] indicated the important role of grain boundaries, where additional variants of martensite nucleate and affect the maximum reversible strain value.

Plastic deformation of NiTi alloys [7, 8] did not cause degradation of the martensitic and R-phase transformation up to 55% deformation [7], while cold rolling of martensitic alloy up to 60% reduction caused the formation of an amorphous structure within shear bands, explained by the mechanical instability against shear stress.

In the present work, CuAlMn alloys containing nearly equal amounts of aluminium and manganese, but various transformation temperatures (A_f above and below room temperature), were deformed by rolling up to 90% to study the degradation of the martensitic transformation, changes of the ordered domain structure and the structure of deformed martensite and of deformation bands.

2. Experimental procedure

Alloys were cast in an induction furnace under an argon atmosphere. The composition of the alloys and the M_s temperatures are given in Table I.

Ingots were hot rolled, quenched from 870 °C to water at 50 °C to avoid stabilization, then cold rolled to reductions from 10%–93%. Structure was examined using a Philips 301 transmission electron microscope at 100 kV. Thin foils were obtained by electropolishing in tenupol 2 at room temperature. Samples for optical microscopy were polished and etched electrolytically in diluted nitric acid. Characteristic transformation temperatures were measured using Du Pont 900 calorimeter.

3. Results and discussion

Fig. 1 shows a set of optical microstructures taken from the cross-section of sheets of alloy B cold rolled to reductions of 0, 10.5%, 31%, 47% and 73%. The initial state is characterized by a fairly large grain size of about 500 μm of β_1 phase (Fig. 1a). At a deformation of 10.5%, samples were already 100% martensitic with large, mostly lens-shaped plates. At the grain boundaries, nucleation of small additional variants may be seen, as observed in bicrystals by Miura *et al.* [6]. Increasing the deformation to 31% (Fig. 1c)

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TABLE I CuAlMn alloy composition (wt%) and M_s temperature

Alloy	Cu	Al	Mn	M_s °C
A	81.0	10.0	9.0	5
B	80.3	9.5	10.2	− 25

causes a refinement of plate structure due to injection of additional variants into plates and internal twinning, as suggested by Adachi and Perkins for CuZnAl [1]. Deformation of 47% causes bending of plates, which clearly indicates activation of slip within martensite and is accompanied by a further refinement of

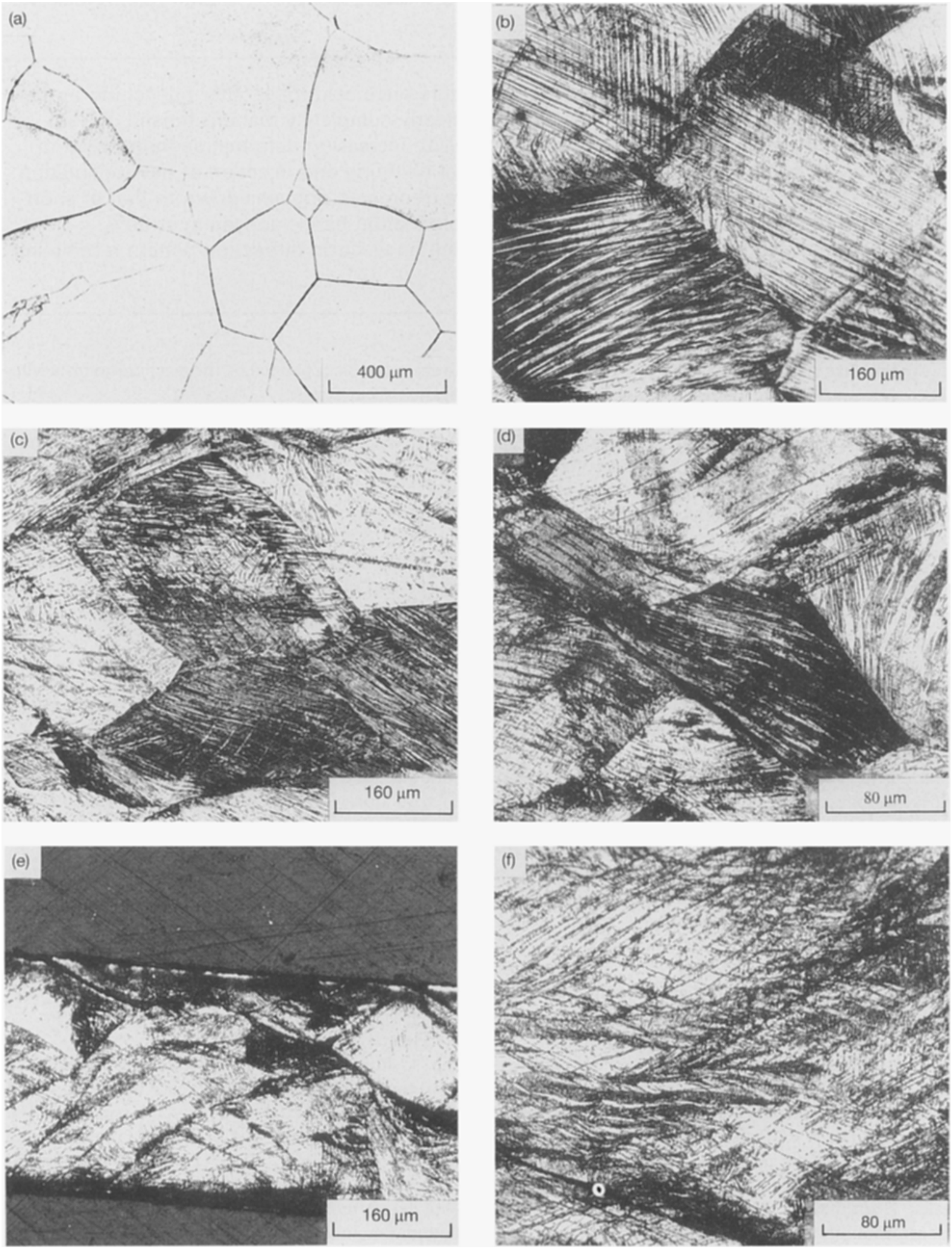

Figure 1 Optical micrographs of alloy B (a) as-quenched, and deformed (b) 10%, (c) 31%, (d) 47%, (e, f) 73%.

plates. The changing shape of the grains can also be seen, indicating passing of shear bands across the grains. At the highest deformation of 73%, shear bands running at an angle of 35 ° to the rolling direction can be clearly seen, as well as a nucleation of cracks within the bands. Martensitic structure is so fine that it can be barely seen. Similar structures were observed in alloy A of higher M_s, where the stress-induced martensite was expected even at lower reductions, as in alloy B, i.e. 10%.

A differential scanning calorimetric (DSC) study of martensitic transformation in deformed alloys did not detect any reverse transformation in samples deformed by only 10%. This is somehow surprising, as CuZnAl alloys show martensitic transformation even at 25% deformation [4, 5], and NiTi at 55% [7, 8]. It may be partially explained by a greater tendency for stabilization of these alloys than that of CuZnAl and NiTi, as can be judged from the quenching and heating experiments. In other words, plastic deformation of investigated alloys stabilizes martensite to such an extent that no reverse transformation occurs. This may be caused by increasing vacancy concentration and dislocation density.

In order to determine changes of plate and dislocation structure, as well as of ordered domain structure, transmission electron microscopy was applied. Fig. 2 shows a set of bright- and dark-field micrographs of alloy A at [0 0 1] γ' zone-axis orientation, deformed to $\varepsilon = 10\%$. Fine equiaxed ordered domains, of about 50 nm in size, can be seen imaged using the 1 1 0 γ' reflection. The bright-field image reveals increased dislocation density, resulting most probably from the basal plane slip. At higher deformation (31%), injection of new martensite variants into the primarily formed plates can be seen (Fig. 3) as reported by Adachi and Perkins [1] for CuAlZn alloys. Streaks in the $\langle 0 0 1 \rangle$ direction increase its intensity due to a higher random stacking faults density and formation

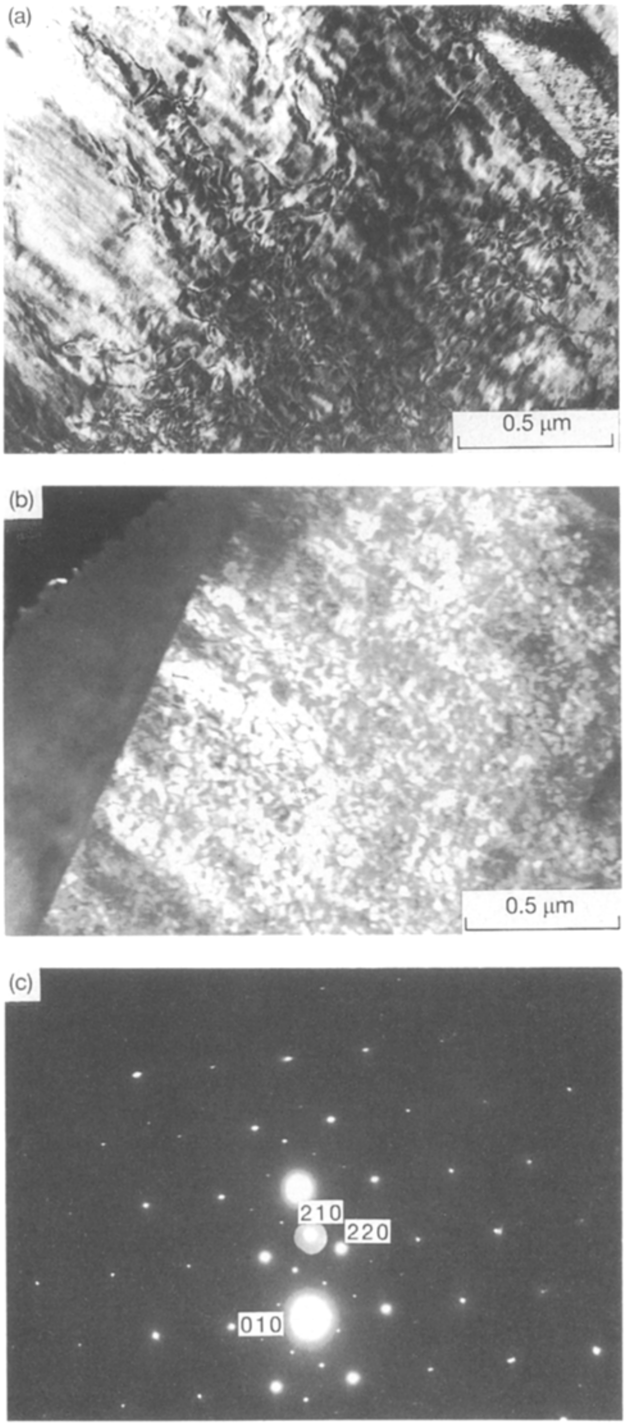

Figure 2 Alloy A deformed 10%: (a) transmission electron micrograph; (b) selected-area diffraction pattern (SADP) showing [0 0 1] γ'_1 orientation; (c) dark-field micrograph taken using 2 1 0 γ'_1 superlattice reflection.

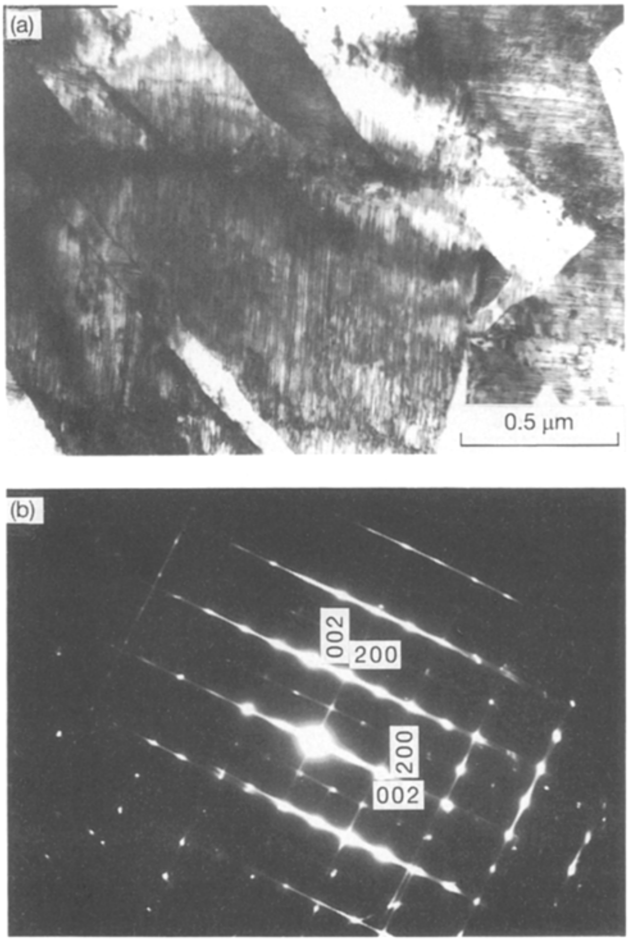

Figure 3 Alloy A deformed 31%: (a) transmission electron micrograph; (b) SADP showing 2 0 2 twins at [0 1 0] γ'_1 zone axis orientations.

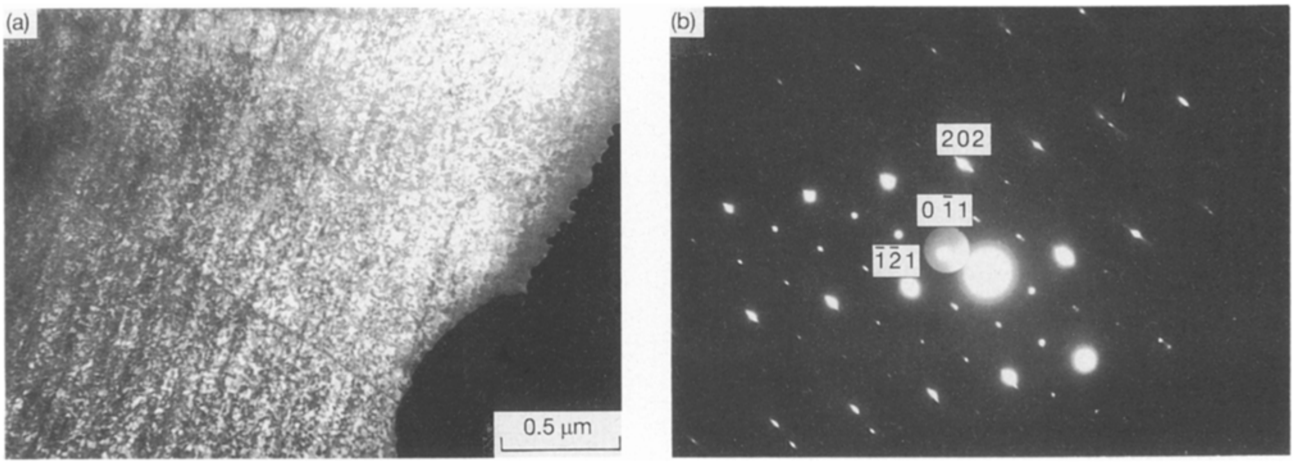

Figure 4 Alloy A deformed 31%: (a) dark-field micrograph taken using 0$\bar{1}$1 superlattice reflection; (b) SADP showing [$\bar{1}$11] γ'_1 zone axis orientation.

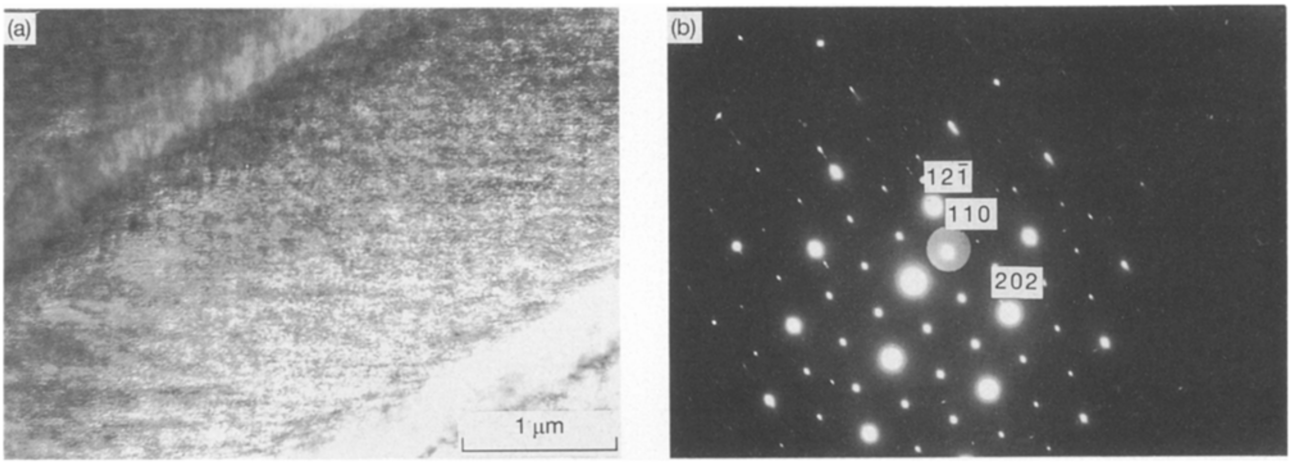

Figure 5 Alloy A deformed 61%: (a) dark-field micrograph taken using 110 superlattice reflection; (b) SADP showing [$\bar{1}$11] γ'_1 zone axis orientation.

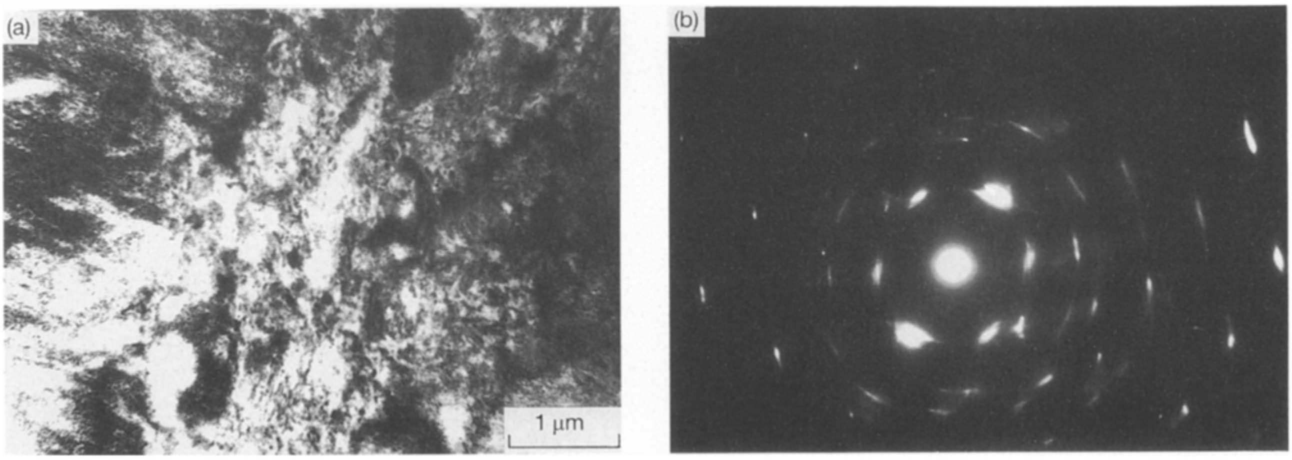

Figure 6 Alloy A deformed 61%: (a) transmission electron micrograph; (b) SADP showing diffused rings from deformation band and reflections from a grain.

of thin layers of other martensite variants, such as 18R within 2H plates. The selected-area diffraction pattern (SADP) shows the directions of twin boundaries and stacking faults, and {202} 2H twins are being formed. No clear reflections from 18R can be seen, being hidden within the streaks, but in other plates they are more pronounced at this orientation. From the contrast of stacking faults in this and other microstructures at [010] orientation, a high density of dislocations can be seen separating the stacking faults.

Fig. 4 shows refinement of ordered domains at this deformation, imaged using 100 γ' reflection. At higher deformation (61%), they are even finer (Fig. 5) and are visible in the form of bright rows which are most probably caused by a small misorientation formed within martensitic plates due to a high concentration of dislocations. It is relatively very small when compared to correspondingly deformed α solid solutions [9], but similar in nature and has an obvious effect on the contrast of ordered domains. In several places, nucleation of deformation bands can be observed at 61% deformation (Fig. 6). They consist of irregular areas of very fine grains, separating martensitic plates showing characteristic elongated subgrain contrast. The diffraction pattern (Fig. 6b) shows reflections from a deformation band diffused along Debye–Scherrer rings and reflections from martensite plates.

At the highest possible deformation (93%) obtained by rolling, both alloys show well-developed deformation bands of extremely small grains (Figs 7 and 8) which give diffraction patterns characteristic of amorphous matter, similar to that reported for NiTi alloys [8]. A slight variation in intensity can be distinguished over the rings, most probably due to the residual texture of microcrystals within bands, but no superreflection rings can be seen and the highest intensity ring corresponds to the atomic nearest neighbour distance, as in the case of the amorphous structure. The fine-grain structure of deformation bands reported in deformed solid solutions [9, 10] never attains such a degree of refinement, as in the case of martensitic alloys. It can be caused by a repeated internal twinning and injection of new variants, in the form of microplates, in the region of deformation bands, where the direction of the flow of the material is forced by the external conditions. The deformation by slip is rather limited due to ordering of marternsite and due to its orthorhombic/hexagonal structure, with limited slip possibilities. Increased dislocation density, subgrain formation and disordering with increasing deformation indicates that slip is, however, also one of the important factors in the martensite deformation.

Fig. 9 shows ordered microdomains after 93% deformation at [010] 2H zone axis orientation. Very small ordered domains can be seen, resembling that of short-range order visible in brighter rows due to plate

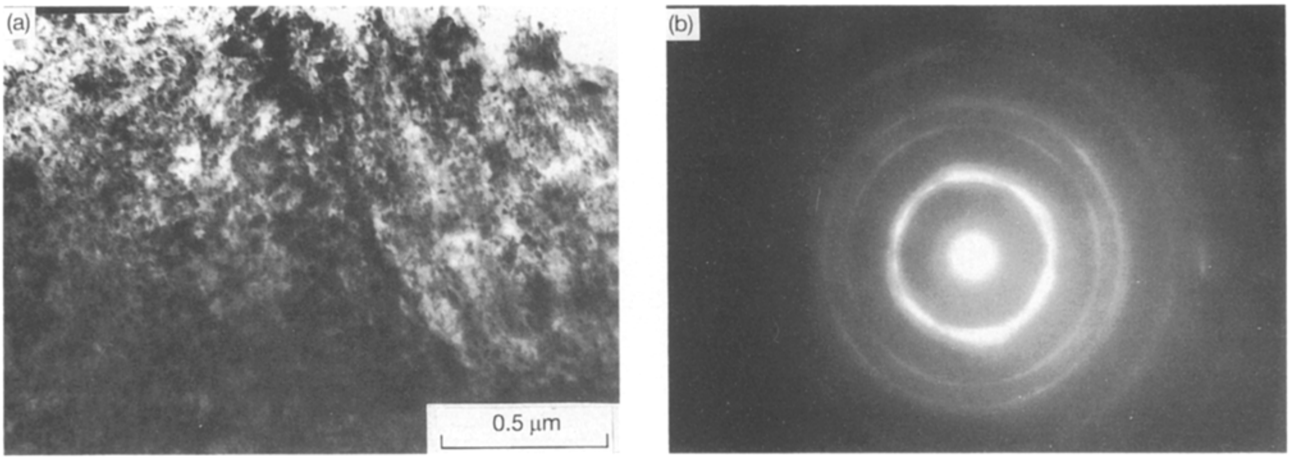

Figure 7 Alloy A deformed 93%: (a) transmission electron micrograph of deformation band area; (b) corresponding SADP.

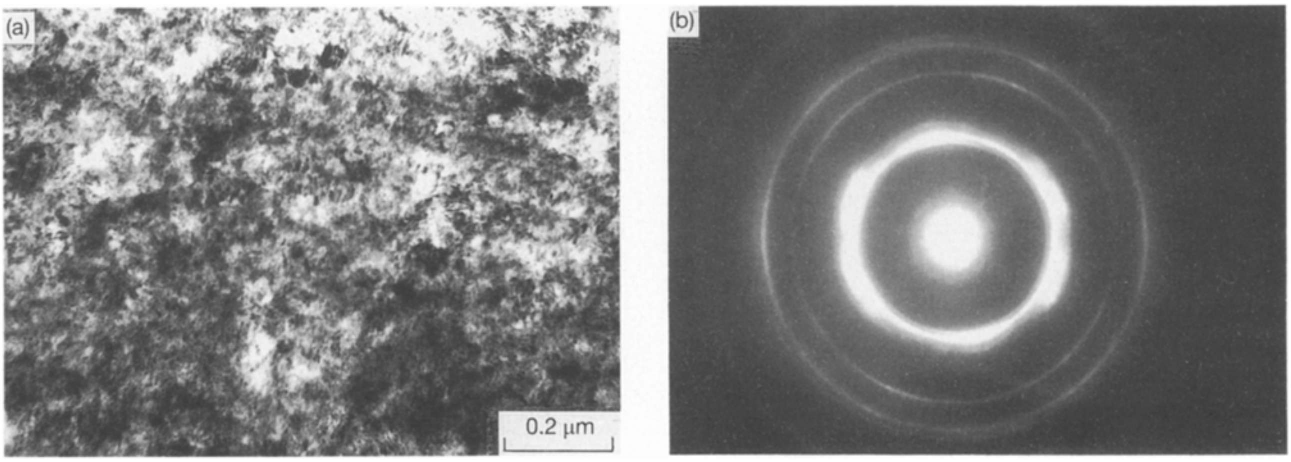

Figure 8 Alloy B deformed 93%: (a) transmission electron micrograph of deformation band area; (b) corresponding SADP.

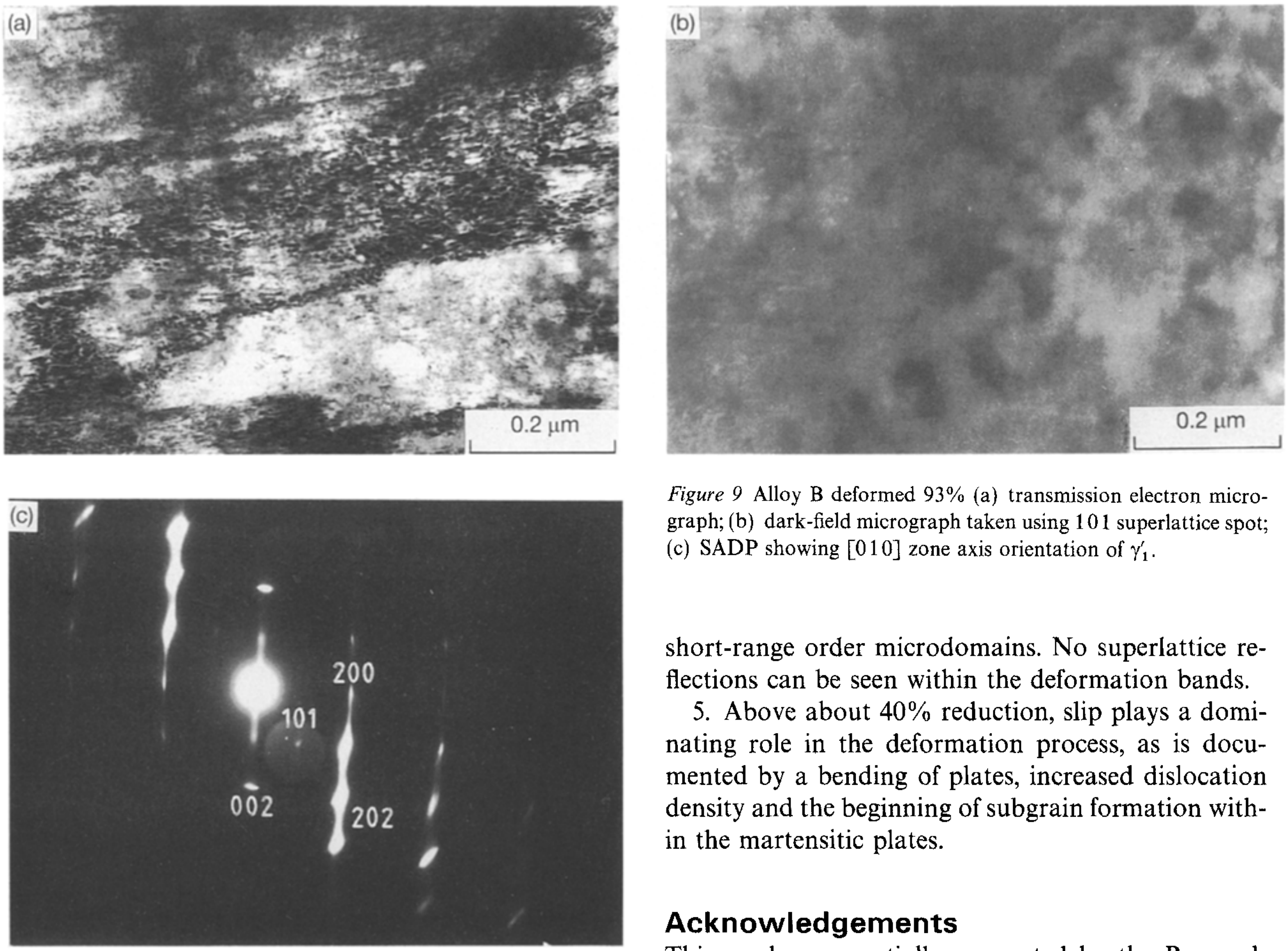

Figure 9 Alloy B deformed 93% (a) transmission electron micrograph; (b) dark-field micrograph taken using 101 superlattice spot; (c) SADP showing [010] zone axis orientation of γ'_1.

misorientation. In the bright field, the change of contrast is due to the presence of elongated subgrains and increased dislocation density.

Conclusions

1. Plastic deformation of 10% of as-quenched β_1 CuAlMn alloys causes complete transformation to martensite, mostly 2H with a small fraction of 18R, and irreversibility of stress-induced martensite.

2. Increasing plastic deformation causes refinement of martensitic plates due to internal twinning and injection of foreign variants into plates.

3. Deformation bands observed first at 61% of deformation increase its size at higher reductions and possess extremely fine grain structure, giving a diffraction pattern resembling that of the amorphous structure.

4. With increasing degree of deformation by rolling, the size of ordered domains of 2H martensitic structure decreases from 50 nm at 10% reduction, to a very small value at 93% reduction, resembling the image of short-range order microdomains. No superlattice reflections can be seen within the deformation bands.

5. Above about 40% reduction, slip plays a dominating role in the deformation process, as is documented by a bending of plates, increased dislocation density and the beginning of subgrain formation within the martensitic plates.

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