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MECHANICAL TWINNING IN $Ti_{50}Ni_{47}Fe_3$ AND $Ti_{49}Ni_{51}$ ALLOYS

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Abstract—Pseudo-twinning and mechanical twinning have been observed in a transmission electron microscopy study of $Ti_{50}Ni_{47}Fe_3$ and $Ti_{49}Ni_{51}$ alloys which have the B2(CsCl) structure. Observation of twinning in ordered alloys is rare and this is the first observation of twinning reported in a B2 structure. The twin planes are the {112} and {114} planes. For {112} pseudo-twins, the composition plane is not the twin plane and the pseudo-twin does not have the B2 structure. For {114} mechanical twins, the composition plane is the twin plane and the twin does have the B2 structure. It is shown that a shear on the {114} plane plus a shuffle of the atoms results in the ordered B2 structure in the twinned region.

Résumé—Nous avons observé du pseudo-maclage et du maclage mécanique au cours d'une étude par microscopie électronique d'alliages $Ti_{50}Ni_{47}Fe_3$ et $Ti_{49}Ni_{51}$ de structure B2 (CsCl). Les observations de maclage dans les alliages ordonnés sont rares et ceci est la première observation de maclage publiée dans la structure B2. Les plans de macle sont les plans {112} et {114}. Pour les pseudo-macles {112}, le plan de composition n'est pas le plan de macle et la pseudo-macle ne présente pas la structure B2. Dans le cas des macles {114}, le plan de composition est le plan de macle et la macle présente la structure B2. Nous montrons qu'un cisaillement dans le plan {114}, plus un réarrangement des atomes, conduit à la structure ordonnée B2 dans la région maclée.

Zusammenfassung—In den Legierungen $Ti_{50}Ni_{47}Fe_3$ und $Ti_{49}Ni_{51}$, die die B2(CsCl)-Struktur aufweisen, wurde die Bildung von Pseudozwillingen und mechanischen Zwillingen elektronenmikroskopisch untersucht. Die Beobachtung der Zwillingsbildung ist in geordneten Legierungen selten; diese Beobachtungen stellen die ersten für eine B2-Struktur dar. Die Zwillingsebenen sind {112} und {114}. Bei den {112}-Pseudozwillingen ist die Ebene der Zusammensetzung nicht die Zwillingsebene; der Pseudozwilling hat auch keine B2-Struktur. Bei den mechanischen {114}-Zwillingen entspricht die Ebene der Zusammensetzung der Zwillingsebene; der Zwilling weist die B2-Strukutr auf. Es wird gezeigt, daß eine Scherung auf der {114}-Ebene und eine Verwerfung der Atome zu einer geordneten B2-Struktur im Zwillingsbereich führt.

INTRODUCTION

It is known that b.c.c. metals may mechanically twin on {112} planes by a simple shear mechanism [1]. However if the same twinning mechanism is applied to materials with the B2 (CsCl) structure the structure is not reproduced because the B2 ordering is destroyed [2]. The result is a new crystal structure, which is also not a mechanical twin since there is no mirror symmetry between the sheared and unsheared regions. As a consequence, mechanical twinning is expected to be difficult in ordered alloys. Some authors have argued that such a shear in an ordered alloy is more accurately described as a stress-induced martensitic transformation rather than mechanical twinning since a new phase is created [2, 3]. However the new phase is created by deformation in response to a shear stress and would not be created by a change in temperature, pressure or composition. If only atomic sites are considered and the atomic type is neglected there is mirror symmetry between the sheared and unsheared region. It has been suggested that such a twin be referred to as a "pseudo-twin" [4]. *A crystal is pseudo-twinned if the atomic sites of the crystal are in a twinned orientation, but not the crystal itself.*

It was pointed out by Laves [5] that, in general, a simple shear of an ordered alloy does not result in a mechanical twin. He also noted that it may be possible for a simple shear plus a shuffle in an ordered alloy to result in a mechanical twin where the sheared and shuffled region has mirror symmetry with respect to the unsheared region. *Following Laves, a twinning mechanism which requires a shear and a shuffle is a complex twinning mechanism, and one which requires only a shear is a simple twinning mechanism.*

Pseudo-twinning has been observed by optical microscopy and Laue diffraction patterns in Fe_3Al, which has the DO_3 structure, when the long range order parameter (S) is less than 0.5 [2]. Pseudo-twinning and detwinning has also been reported [6] in Fe_3Be which also has the DO_3 structure. However this was based on observations of surface relief in the optical microscope and therefore it is possible that the observations are of stress-induced martensite rather than twinning. Mechanical twinning has never been reported in the B2 structure.

This paper reports the direct observation of pseudo-twinning and mechanical twinning in TiNi alloys with the B2 structure. Simple and complex twinning mechanisms by which mechanical twinning may occur in the B2 structure are presented.

EXPERIMENTAL

(I) Alloy preparation

Alloys with nominal composition of $Ti_{50}Ni_{47}Fe_3$ and $Ti_{49}Ni_{51}$ were used. The cast ingots were hot swaged and then rolled into sheets 0.6 mm thick. Tensile samples were stamped from the sheet, annealed at 850°C and air cooled. The dimensions of the gage region are approximately 0.6 mm × 6 mm × 80 mm.

TiNi alloys undergo a martensitic transformation from the B2 structure to a monoclinic or distorted B19 structure [7]. The reversibility of this martensitic transformation is responsible for their shape-memory properties. M_s, the temperature at which martensite starts to form on cooling, was found to be $-140°C$ for the $Ti_{50}Ni_{47}Fe_3$ alloy and $-40°C$ for $Ti_{49}Ni_{51}$.

(II) Mechanical deformation

The alloys were plastically deformed using two methods, by isothermal simple tension and by thermally cycling through the martensitic transformation while under a constant load.

(A) Tensile test. A $Ti_{50}Ni_{47}Fe_3$ tensile sample was deformed to 10% strain and a $Ti_{49}Ni_{51}$ sample was deformed to 8% strain with a strain rate of $2.2 \times 10^{-3}\,s^{-1}$ at 150°C. This temperature is well above the temperature at which martensite may be stress-induced in either alloy indicating that the samples had undergone a large plastic strain.

(B) Thermal cycling under a load. The original purpose of these experiments was to determine the microstructure of an alloy that had been thermally cycled under a high load. A $Ti_{50}Ni_{47}Fe_3$ sample was placed under a tensile stress of 500 MPa. and cycled in temperature from 125° to $-190°C$ and back to 125°C. A $Ti_{49}Ni_{51}$ sample was thermally cycled between the same temperatures as the $Ti_{50}Ni_{47}Fe_3$ sample except with a tensile stress of 400 MPa. The samples were thus cycled through the martensite transformation while under a load.

(III) Transmission electron microscopy

Transmission electron microscopy (TEM) specimens were prepared by sparkcutting 3 mm discs from the gage region of the deformed samples, mechanically thinning the discs to 100 microns using 400 grit SiC paper and electropolishing in a 8% perchloric acid–92% acetic acid electrolyte at room temperature and 25 volts. The TEM experiments were performed in a Philips EM400 using a double-tilt stage.

RESULTS AND DISCUSSION

(I) General observations

Twins with {112} and {114} twin planes were observed in both alloys whether deformed in a tensile test or by thermal cycling under a constant load. The crystallographic observations from both alloys are similar and are analyzed together. Approximately 50% of the volume of the thermally cycled samples was twinned while there was less than 10% of the volume twinned in the samples pulled under tension, despite the fact that the maximum stress and strain were lower in the case of thermal cycling. Therefore more of the observations are from the thermally cycled samples. Figure 1 shows the twinned

Fig. 1. Bright field image of twinned microstructure from a $Ti_{50}Ni_{47}Fe_3$ alloy thermally cycled under a stress of 400 MPa.

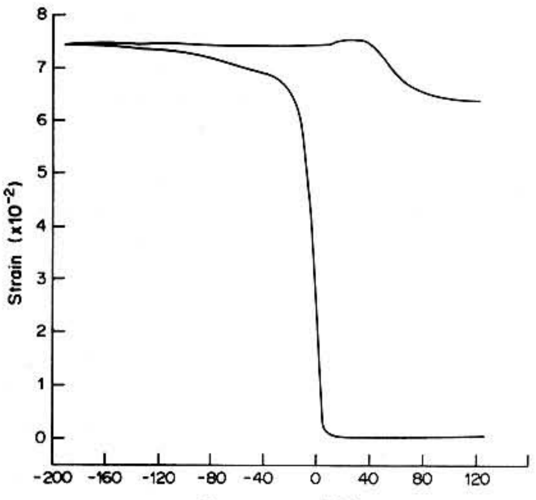

Fig. 2. Strain vs temperature for $Ti_{49}Ni_{51}$ sample thermally cycled under a stress of 500 MPa.

microstructure of the thermally cycled $Ti_{50}Ni_{47}Fe_3$ alloy. The extremely fine dimensions of the structure are evident and electron diffraction indicates that there is no martensite present.

Analyses of 10 separate observations of {112} pseudo-twins were made and in only one case was the composition plane the same as the twin plane. The composition plane is the plane of contact between the twinned regions. In four cases the composition plane was a {112} plane but a different {112} plane from the twin plane. Finally in the other five cases of {112} twinning the composition planes were found to vary among a wide range of planes. Six observations of {114} twins were analyzed and in all six cases the composition plane was the twin plane. It is not clear as to why the composition plane differs from the twin plane for {112} twins.

Figure 2 shows the strain as a function of temperature for the $Ti_{49}Ni_{51}$ alloy thermally cycled under a constant load. Upon cooling there is a large strain as the austenite transforms to martensite under a load. This large strain is accommodated by the austenite to martensite transformation because of the different shape of the martensite unit cell. Upon heating 6.3% strain is not recovered during the reversion to austenite and must therefore be accommodated by twinning and slip. The martensite may transform to austenite and then twin or the martensite may transform directly to a twinned austenite microstructure. It is not clear at present as to the actual sequence of events. However the observation that samples deformed in simple tension have a much lower twin density indicates that the martensite transforms directly to a twinned austenite microstructure upon heating under a load.

(II) {112} twinning

The structure of {112} pseudo-twins in a B2 structure has been theoretically analyzed by Cahn and Coll [2]. They pointed out that a simple shear on {112} planes will produce a new structure which is tetragonal with $a = b = \sqrt{2}a_{B2}$ and $c = a_{B2}$ where a_{B2} is the B2 lattice parameter. The ratio of AB, AA and BB nearest neighbors for this new structure is exactly that for a disordered alloy. In the $[1\bar{1}0]$ projection shown in Fig. 3 the {112} pseudo-twin is seen where $K_1 = (112)$, $\eta_1 = [\bar{1}\bar{1}1]$, $K_2 = (11\bar{2})$, $\eta_2 = [111]$. The

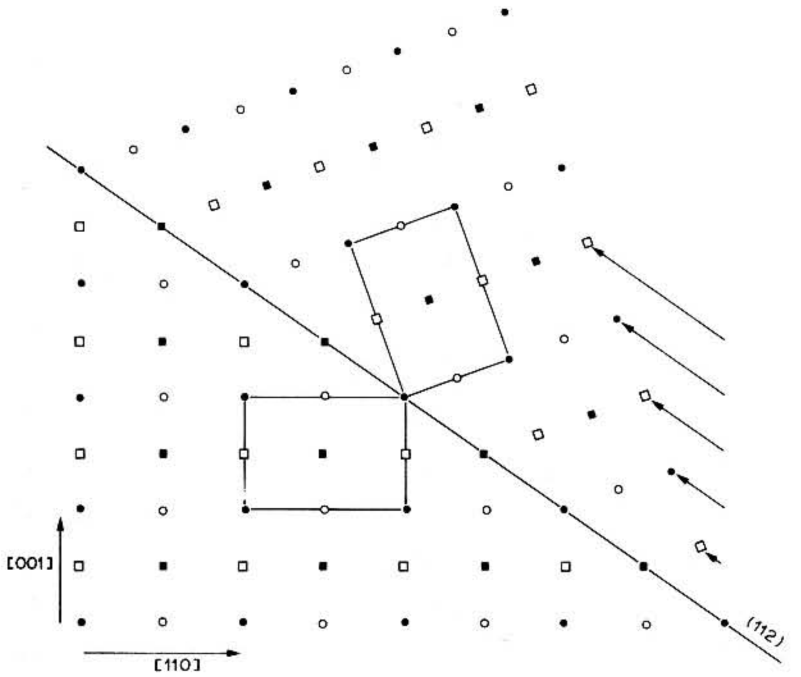

Fig. 3. $[1\bar{1}0]$ projection of {112} pseudo-twin in the B2 structure. The circles and squares refer to different types of atoms. The open symbols are atoms out of the plane and the filled symbols are atoms in the plane.

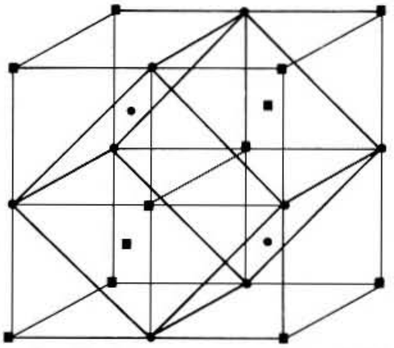

Fig. 4. Unit cell of structure created by {112} pseudo-twinning shown in Fig. 2.

magnitude of the twinning shear is 0.707 and is identical to that for b.c.c. twinning. Figure 4 is a perspective drawing of the tetragonal unit cell of the structure formed by a simple shear on a {112} plane as shown in Fig. 3. The B2 structure is not preserved in the sheared region.

Figure 5 is a [1$\bar{1}$0] projection where the twinning shear is twice as large and in the opposite direction to the shear in Fig. 3. This results in a sheared region with the B2 structure. Therefore this is a simple mechanism for mechanical twinning in a B2 structure. Treating the atoms as hard spheres the shear shown in Fig. 5 would be more difficult than the shear shown in Fig. 3 because the distance of closest approach is smaller.

Laves pointed out that if the shear shown in Fig. 3 is produced, followed by a shuffle of every other (112) plane in the sheared region by the nearest-neighbor distance, a mechanical twin with the B2 structure is produced [5]. Laves defined a variable Q which is a quantitative measure of the complexity of the shuffle in a complex twinning mechanism [5]. Let r be the distance an atom has to shuffle divided by the nearest neighbor distance. Then Q is simply the average value of r over all the atoms. For this complex mechanism, Q equals 0.5.

For {112} pseudo-twinning in TiNi, the B2 superlattice reflections are not observed in the twin diffraction pattern indicating a loss of ordering. Figure 6(a) is a [110] zone axis electron diffraction pattern showing {112} pseudo-twinning in the $Ti_{49}Ni_{51}$ alloy thermally cycled under a constant load. Figure 6(b) is a schematic drawing of Fig. 6(a). The weak double diffraction spots in Fig. 6(a) have been omitted from Fig. 6(b). Figure 6(c) is a dark field image from the {112} twin reflection. The micrographs are correctly oriented with respect to the diffraction pattern. In this case it can be seen that the habit plane of the {112} twin is (110) and that the B2 superlattice reflections are not twinned by the {112} twin.

The twin pattern shown in Fig. 6(a) does not correspond to the pattern calculated from the tetragonal structure shown in Fig. 4. The tetragonal structure, in addition to not producing the B2 super-

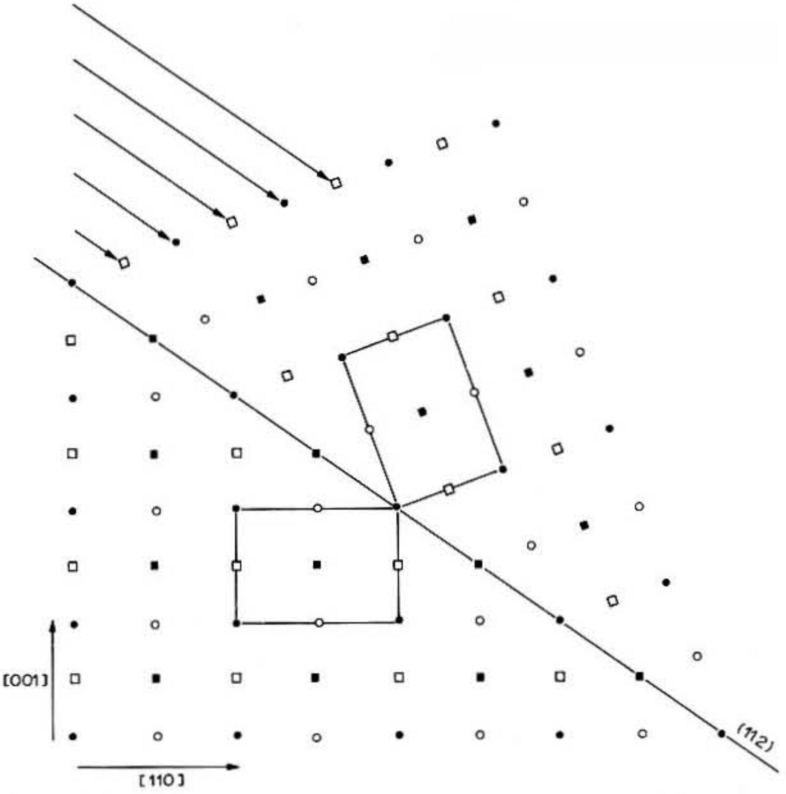

Fig. 5. [1$\bar{1}$0] projection of shear on the (112) plane which preserves the B2 structure.

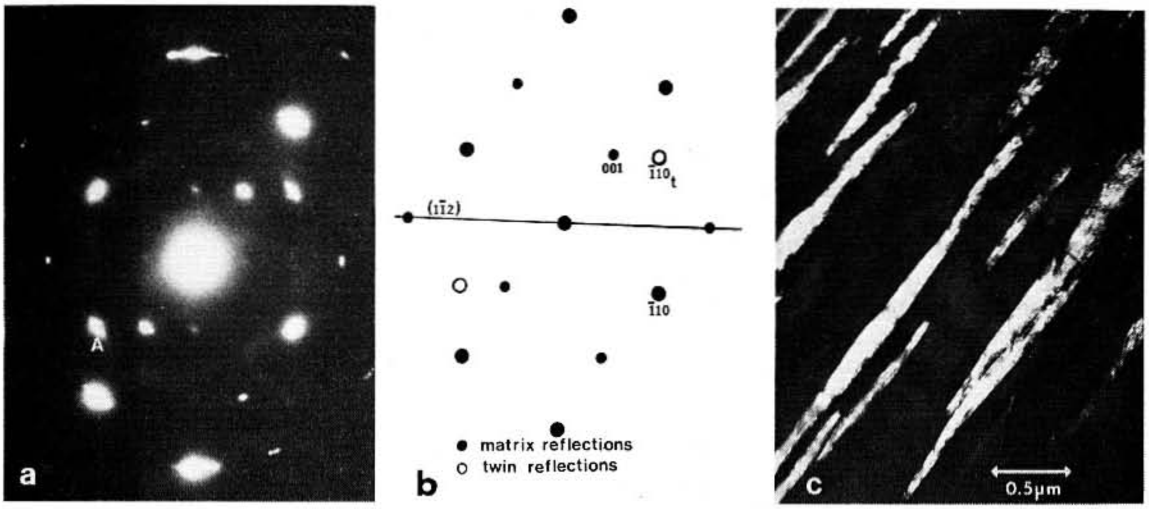

Fig. 6. (a) [110] zone axis showing {112} twin in a $Ti_{49}Ni_{51}$ alloy thermally cycled under a stress of 500 MPa. (b) Schematic drawing of (a) identifying reflections. (c) Dark field from twin reflection A.

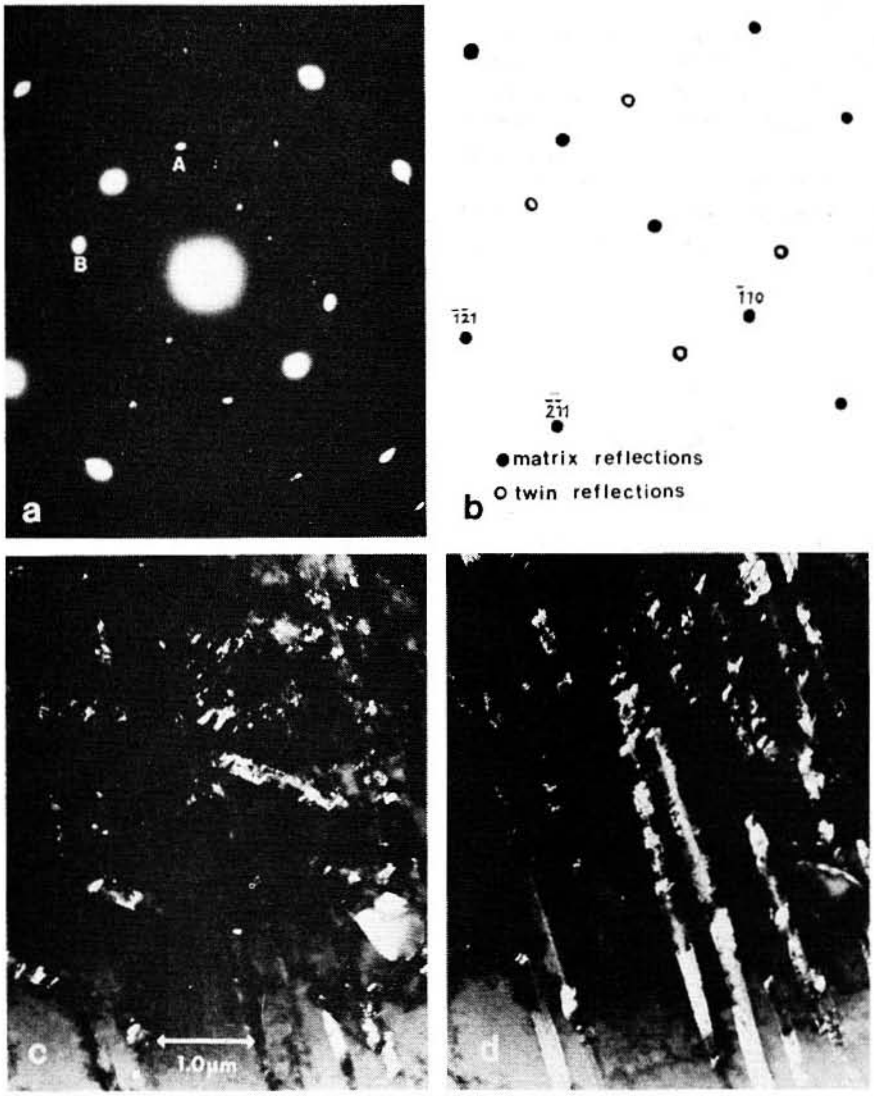

Fig. 7. (a) [113] zone axis electron diffraction pattern showing {112} pseudo-twinning in a $Ti_{50}Ni_{47}Fe_3$ alloy thermally cycled under a stress of 400 MPa. (b) Schematic drawing of (a) identifying reflections. (c) Dark field from twin reflection A. (d) Dark field from twin reflection B.

lattice reflections, should give rise to reflections, which are not observed, at one-half the distance of the (110) reflections. Therefore it is not clear which of the two possible mechanisms above are operative, or whether another mechanism has occurred.

Figure 7(a) is a [113] zone axis electron diffraction pattern showing {112} pseudo-twinning in a $Ti_{50}Ni_{47}Fe_3$ alloy. Figure 7(b) is a schematic drawing of the diffraction pattern shown in Fig. 7(a). The twin planes are $(1\bar{2}1)$ and $(2\bar{1}1)$ planes. The $(1\bar{2}1)$ twin has a $(2\bar{1}1)$ composition plane and the $(2\bar{1}1)$ twin has a $(2\overline{13}5)$ composition plane. Dark field images from the twin reflections are shown in Fig. 7(c) and 7(d).

The order parameter of TiNi alloys is not known. FeAl alloys in the B2 structure have an order parameter close to unity regardless of temperature from which the alloy is quenched [2]. We believe that TiNi alloys are similar because of the limited stoichiometric range of the B2 phase [8]. Therefore the mechanical twinning observed in TiNi is not believed to be due to a lack of long range order.

(III) {114} twinning

Figure 8 is a $[1\bar{1}0]$ zone axis electron diffraction pattern showing a {114} twin, in the $Ti_{49}Ni_{51}$ alloy thermally cycled under a constant load, and the B2

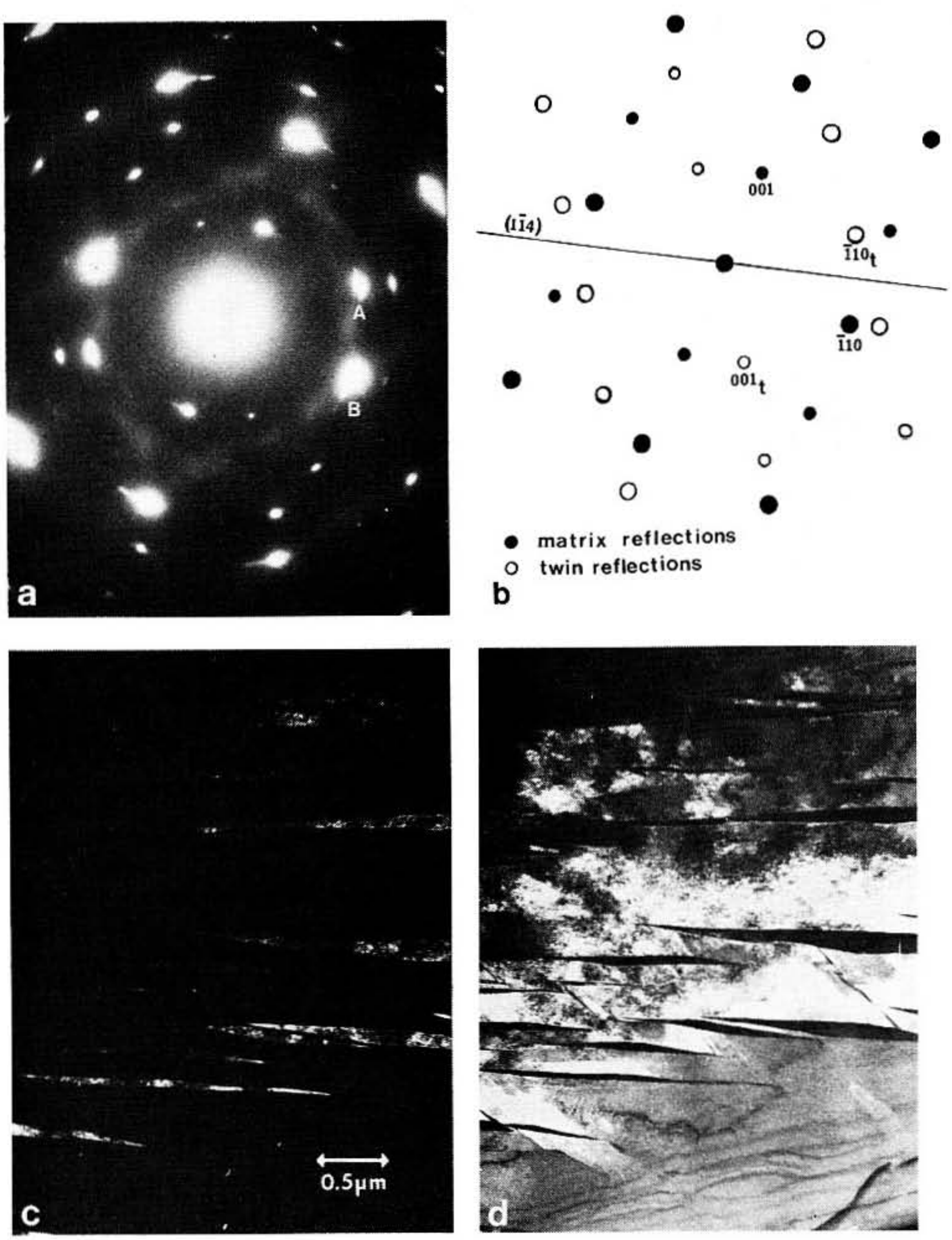

Fig. 8. (a) [110] zone axis showing {114} twins. (b) Schematic drawing of (a) identifying reflections. (c) Dark field from twin reflection A. (d) Dark field from matrix reflection B.

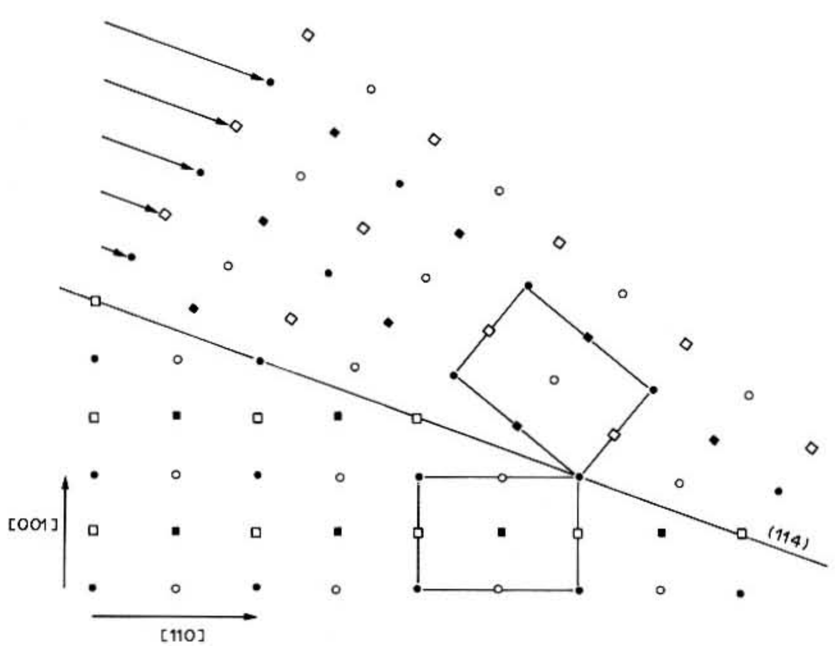

Fig. 9. [1$\bar{1}$0] projection of structure created by simple shear on the {114} plane. Symbols are defined as in Fig. 3.

superlattice reflections are twinned. In all the observations of {114} twinning the B2 superlattice reflections are present in the twin pattern. Therefore the {114} twinning does result in the ordered B2 structure.

A simple shear on the {114} plane produces a new structure which will be different from the structure created by {112} pseudo-twinning. Figure 9 is a [1$\bar{1}$0] projection of a shear in the (114) plane, the magnitude of the twinning shear being 0.707. Figure 10 shows the unit cell of the structure created by this shear. The nearest-neighbor distance in this structure is much closer than that in the B2 structure and therefore it is not expected to be stable. If the structure in Fig. 10 undergoes the shuffle shown by the arrows in Fig. 11 the B2 structure results. The required shuffle may be described as a shuffle of every other {110} plane by a distance one-half of a_{B2} in the ⟨001⟩ direction. It has a component normal to the twin interface and can be accommodated at the interface by rows of vacancies. The shuffle is expected to occur in the direction away from the interface since the atoms at the interface after the shear are separated be a distance less than the nearest-neighbor distance. A [1$\bar{1}$0] projection of the interface after the shuffle is shown in Fig. 12.

The rows of vacancies on alternating (110) planes at the twin interface shown in Fig. 12 implies rows of interstitials at the advancing twin interface. This may be accommodated by rows of vacancies on the other set of alternating (110) planes at the advancing twin interface. Figure 13 shows such a (114) twin platelet where the twinned region retains the B2 structure. The creation of the (114) twin platelet requires a slight dilation. Therefore the stress to form twins should depend on the hydrostatic pressure. However once this platelet is nucleated, growth may occur by continuous shear and shuffle or (114) planes. For {114} twinning r is zero for half of the atoms and r equals $\sqrt{1/3}$ or 0.5774 for the other half of the atoms.

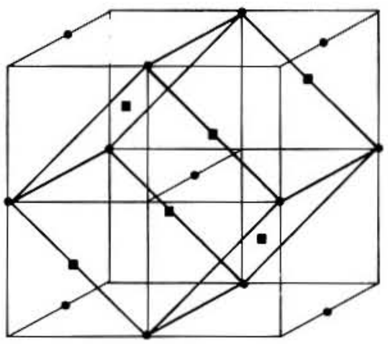

Fig. 10. Unit cell of structure created by simple shear on {114} plane.

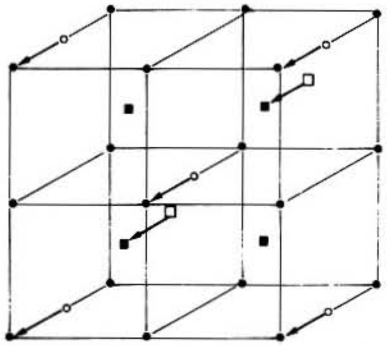

Fig. 11. Shuffling required to bring structure in Fig. 10 to the B2 structure.

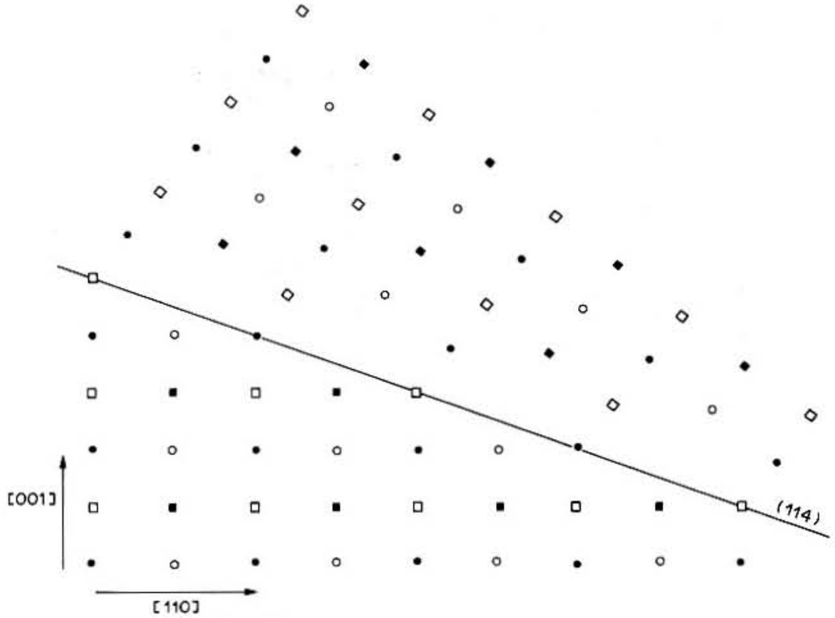

Fig. 12. [1$\bar{1}$0] projection of the (114) twin after shuffle.

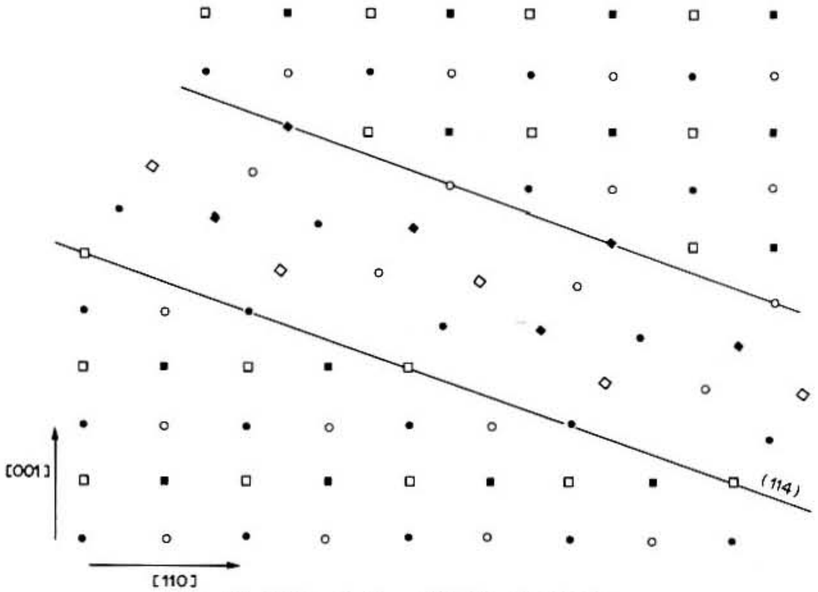

Fig. 13. [1$\bar{1}$0] projection of (114) twin platelet.

Therefore Q equals 0.2887 for the shuffle to create {114} twins. This is a lower value for Q than the shuffle for the (112) twins. Therefore the shuffle to create (114) twins is expected to be easier than the shuffle for (112) twins.

It is important to point out that the exact mechanism by which mechanical twinning on {114} planes occurs is not known and the shear and shuffle steps mentioned are to illustrate that mechanical twinning on {114} planes in response to a shear stress can result in a twin with the B2 structure. As mentioned earlier the twinned B2 structure may be formed directly from the martensite structure upon heating under a load.

A {114} twin orientation may result from double twinning on {112} planes [9]. However, since the observed composition plane is the twin plane, this is not believed to be the case.

Finally it should be pointed out that the underlying reasons for these phenomena in TiNi alloys are still unknown. Similar observations have not been reported on other alloys with the B2 crystal structure. For thermally cycled samples, the possible influence of the martensitic phase transformation and the lattice instability of TiNi cannot be ruled out.

CONCLUSIONS

(1) TiNi alloys with a B2 structure can deform by mechanical twinning and pseudo-twinning.

(2) Thermal cycling under a constant load produces a high density of mechanical twins and pseudo-twins.

(3) Pseudo-twins are observed with {112} twin planes, with the B2 ordering destroyed in twinned regions.

(4) Mechanical twins are observed with {114} twin planes where the B2 structure is retained.

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