HREM Studies on the Microstructure of Severely Cold-Rolled TiNi Alloy after Reverse Martensitic Transformation

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Abstract The microstructure of Ti-49.8at.\%Ni alloy, which was cold rolled to about 30\% reduction in thickness in its martensitic condition and subsequently heated up to 200°C for half an hour, has been studied by high resolution electron microscopy. The interface between parent phase and martensite is not smooth and well coherent. The boundary between two subgrains of the parent phase is not straight but perfectly coherent, with partial dislocation observed at the interface. Inside some parent phase grains, thin plate-like \{114\} and spear-like \{112\} twin-related parent phase variant pairs are observed. The \{114\} twinning boundary is relatively straight, but with two or three atomic-height blurred layers existing near the interface.

1. Introduction

Recently, the effect of cold rolling on the martensitic transformation and microstructure of near equiatomic TiNi alloy have been intensively studied [1-3] through TEM, IF, DSC and XRD. Some studies[4-5] have been concerned with the influence of annealing treatment right after the cold rolling on the shape memory effect and pseudoelasticity characteristics of TiNi alloy. However, there has been no systematic report about the microstructure of cold-worked martensitic TiNi alloys after annealing treatment at different temperatures, especially that subjected to a reverse martensitic transformation at temperature below 300°C.

2. Experimental procedure

A Ti-49.8 at.\%Ni alloy was prepared by consumable arc-melting under an Ar atmosphere in a water-cooled copper crucible. The ingot was hot swaged and rolled at 850°C to strips of approximately 5mm thickness. After annealed at 850°C for 1 hour and followed by cooling with the furnace, the specimens were quenched into the liquid nitrogen to achieve full martensite structure, then cold-rolled at room temperature to the extent of 30\% reduction in thickness step by step. Subsequently, the specimen was annealed at 200°C for half an hour. The foils for HREM observations were mechanically polished to 50 μm and ion thinned. HREM observations were performed by a JEOL-2000EX II electron microscope operated at 200 kV using a top-entry type double-tilt specimen stage with angular ranges of ±10°.

3. Results and discussion

HREM observation shows that the majority of the microstructure in the specimen are parent phases, with a small amount of retained martensites coexisted. This microstructural condition could be easily understood to be the result of martensite stabilization. Fig.1 shows the high resolution image of the dual phases microstructure. It could be noticed that the parent phase–martensite interface is not smooth but well-coherent in Fig.1. on the upper right side of
which these two phases interlace each other, exhibiting broad strain field contrast. Some degree of recovery occurs in the cold-worked specimen after annealing at low temperature. Fig. 2 shows the HREM image of the boundary between two parent phase subgrains. with the

Fig. 1 HREM image of the boundary between the parent phase and the retained martensite. Electron beam // [11 1] // [1 1 0] _M

Fig. 2 HREM image of the boundary between two subgrain in the wholly austenited area and its corresponding EDPs. Electron beam // [11 1] _P
corresponding EDPs superimposed. The boundary is not very straight and perfectly coherent, with a relatively strong strain contrast observed near it. A partial dislocation can be noticed in Fig.2, as marked by a “⊥” symbol. The difference of the orientation between the two parent phase grains is about 4 to 5°, indicative of subgrains rather than a random, recrystallized, fine grain structure.

Some parent phase variants could be found inside individual grains. Fig.3(a) shows the typical spear-like morphology of parent phase variants, in which the thin plate variant coupling A and B are found to be {114} twin related whereas the spear-like variants pair A and C could be indexed to be {112} twin related, as obviously indicated by the corresponding EDPs shown in Fig.3(b). The variants B and C could also regarded as {114} twin related variant coupling, since their conjunction planes are (1̅ 0̅ 4)B and (114)C, which are from the same family in the B2 structure. Fig.3(c) illustrates the HREM image of the (114) intervariant boundary. It is relatively straight, with two or three atomic layers height blurred regions existing near the interface, like a midrib.

Fig.3. (a). Typical morphology of parent phase twin-related variants inside individual grain; (b). Corresponding EDPs taken from the variants A, B and C in (a). Electron beam // [1̅ 10]p; (c). HREM image of the {114} twinning boundary. Electron beam // [1̅ 10]p.
The incident electron beam is parallel to \( [\bar{1}10] \), zone axis in Fig.3, from which direction the schematic diagram of the relative atomistic orientations between (114) twin-related variants was given by Goo et al.[6]. As discussed by Goo, twinning on (114) plane involves atoms undergoing a shear strain of 0.707 in the [221] direction. In order to preserve the \( B_2 \) structure, Goo’s model involves half the atoms, on alternating (110) planes, shuffling 0.5a, in the [001] direction. Clearly, two types of atomic matching might exist at the (114) twinning boundary. These special atomic configurations at twinning boundary might cause the appearance of this midrib featured interfacial structure.

The \{114\} and \{112\} twins were found to be formed by deformation of the \( B_2 \) parent phase without the occurrence of stress induced martensitic transformation, and believed to be mechanical twinnings in Ti\(_{50}\)Ni\(_{50}\)Fe\(_4\) and Ti\(_{50}\)Ni\(_{45}\) alloys[6-7]. For the present specimen annealed above the recrystallization temperature, our recent study show that no twinning substructure is observed inside the parent phase grain at room temperature. According to Koike et al.[3], the dislocation density had been estimated to be about 1-5\times10^{13}/cm\(^2\) in the local area in the 30% reduced TiNi alloy. These dislocation configurations must have important influence on the microstructural aspect in the specimen with different post annealing treatment. It can be suggested that for the present low temperature annealed specimen some dislocations would die out during the reverse martensitic transformation while others would retain even in the newly-formed parent phase. Might these residual unrecoverable defects during the reverse transformation from the severe cold-rolled martensite to parent phase be responsible to the formation of the above several kinds of parent phase twinning variants. They would help accommodating the twin formation and the associated shuffle, rather than being a hindrance, when the Burger vector of the slip dislocations are in the same directions as the shuffle to realize mechanical twinning.

4. Conclusions

(1) The parent phase-martensite interface is not smooth but well-coherent.
(2) The boundary between two subgrains of the parent phase is not straight and perfectly coherent, with partial dislocation observed at the interface.
(3) Inside some parent phase grains, \{114\} and \{112\} twin-related variant pairs are observed. The \{114\} twinning boundary is relatively straight, with two or three atomic-height blurred layers existing near the interface. The residual unrecoverable defects during the reverse transformation from the severe cold-rolled martensite to parent phase might be responsible to the formation of these twinning variants.

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References


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