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TEM investigation of adaptive martensite occurring in ultrafine-grained ferromagnetic Ni-Mn-Ga shape memory alloys

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Ni-Mn-Ga alloys belong to a new class of ferromagnetic shape memory alloys that can show large and reversible magnetic field-induced strains. The phase stability and thus the functional properties of ferromagnetic shape memory alloys sensitively depend on their chemical composition. The $L2_1$ Heusler austenite of Ni-Mn-Ga alloys can transform to different martensitic structures including the non-modulated (NM) tetragonal lattice as well as modulated structures such as 6M, 10M and 14M [1]. In addition, crystal size at the nanoscale can significantly impact the formation of thermoelastic martensite [2,3]. It is the aim of the present paper to study the impact of grain size on the martensitic transformation of $Ni_{54}Mn_{25}Ga_{21}$ high temperature shape memory alloys.

$Ni_{54}Mn_{25}Ga_{21}$ alloys (prepared by induction melting in inert gas) were subjected to high pressure torsion followed by heating to 500°C to achieve an ultrafine-grained structure in the bulk material. Thin foils for transmission electron microscopy (TEM) were prepared by mechanical grinding followed by dimpling and ion milling. The TEM analysis was carried out using lattice fringe images and selected area diffraction (SAD) of small grains carefully tilted to specific beam directions (BD).

In their coarse grained state, $Ni_{54}Mn_{25}Ga_{21}$ alloys showed a martensitic phase transformation from the austenitic Heusler phase to the NM martensite. Contrary to that, in the case of the ultrafine grains (mean grain size about 140 nm) the austenite transformed to the modulated 14M martensitic structure. This is discussed using the concept of adaptive martensite i.e. nanotwins of the NM martensite that occur with a twinning periodicity of 7 atomic layers and a 2/5 ratio of the twin widths forming the metastable 14M martensitic structure [4,5].

Figures 1(a)-(c) show TEM lattice fringe images obtained by tilting the grains to the [201] zone axis of the 14M martensite and allowing the weak $\pm[020]$ satellite reflections to pass the objective aperture together with the transmitted beam (cf. the SAD pattern shown in Figure 1(d)). Fringes with a spacing of 1.48±0.02 nm dominate reflecting the seven layered $(\bar{5}2)_2$ stacking sequence expected in the case of a 14M adaptive martensite. However, frequently the periodicity of the contrast oscillations was interrupted; stripes of dark or bright contrast were encountered (e.g. near A in Figure 1(b) and (c)) that had a width larger than caused by the 14M martensite. Consequently, these stripes corresponded to larger twinning periodicities of the NM martensite. Near B in Figure 1(c), also rather thick twin variants of NM martensite were observed. This agrees with the concept of adaptive martensite where NM and 14M can coexist over a broad temperature range [5]. In the ultrafine grains, minimization of the transformation strain energy is most effectively facilitated by nanoscale twinning [2,3]. Therefore in ultrafine grained $Ni_{54}Mn_{25}Ga_{21}$, nanoscale twinning of the NM martensite yields the formation of the 14M lattice structure.

While most of the ultrafine grains contained a single variant of 14M martensite, some grains were also encountered that contained two twin related variants of the adaptive martensite (cf. Figure 2). The two variants were rotated by an angle of about 125°C around a common [201] zone axis and match at a common $(\bar{1}72)$ interface (indicated by dashed lines in the lattice fringes images of Figures 2(a) and (b)). These junction planes were rather straight and sharp (width less than 2 nm); each of the variants shows contrast oscillations frequently deviating from the perfect $(\bar{5}2)_2$ stacking sequence similar to those observed in grains containing a single variant of the martensite only (cf. Figures 1(b) and (c)). Based on the analysis of Figure 2, the twinning of the adaptive martensite was of type I. Twins with a similar crystallographic relationship were also observed in coarse grains of Ni-Mn-Ga alloys [6].

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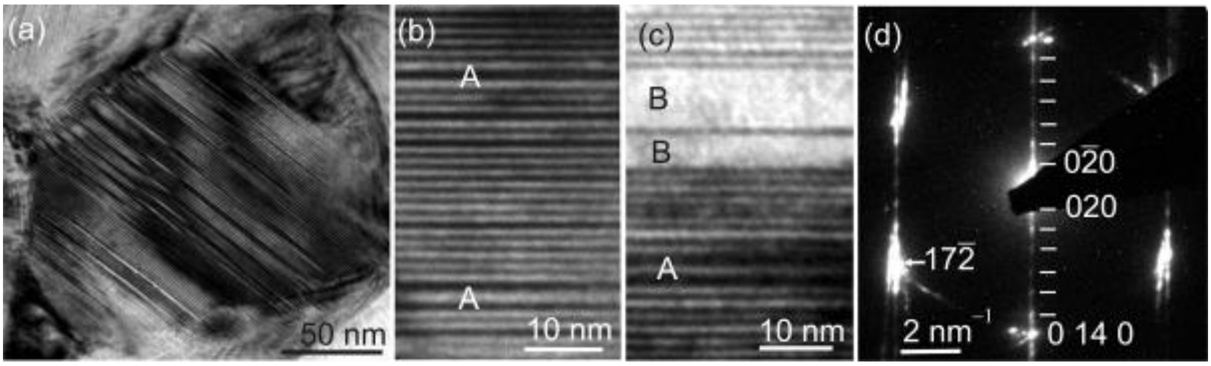

Figure 1. Ultrafine grained $Ni_{54}Mn_{25}Ga_{21}$. (a) TEM lattice fringe image of a grain containing a single variant of 14M martensite. (b) and (c) Contrast oscillations with a spacing of 1.48 nm arise by a 7 layer sequence of nanotwins of the NM martensite (corresponding to the $(\bar{5}2)_2$ stacking sequence of adaptive 14 martensite). These oscillations are frequently interrupted by oscillations with larger periodicities (near A). Areas with uniform contrast (near B) correspond to rather thick twins of the NM martensite. (d) SAD pattern. Six weak satellite reflections (indicated by lines) occur between the primary beam and the main ±[0 14 0] reflections. These satellite reflections are characteristic for the modulated 14M martensite. Weak streaks running parallel to [0 14 0] are caused by the stacking disorder. (**BD** = [201]).

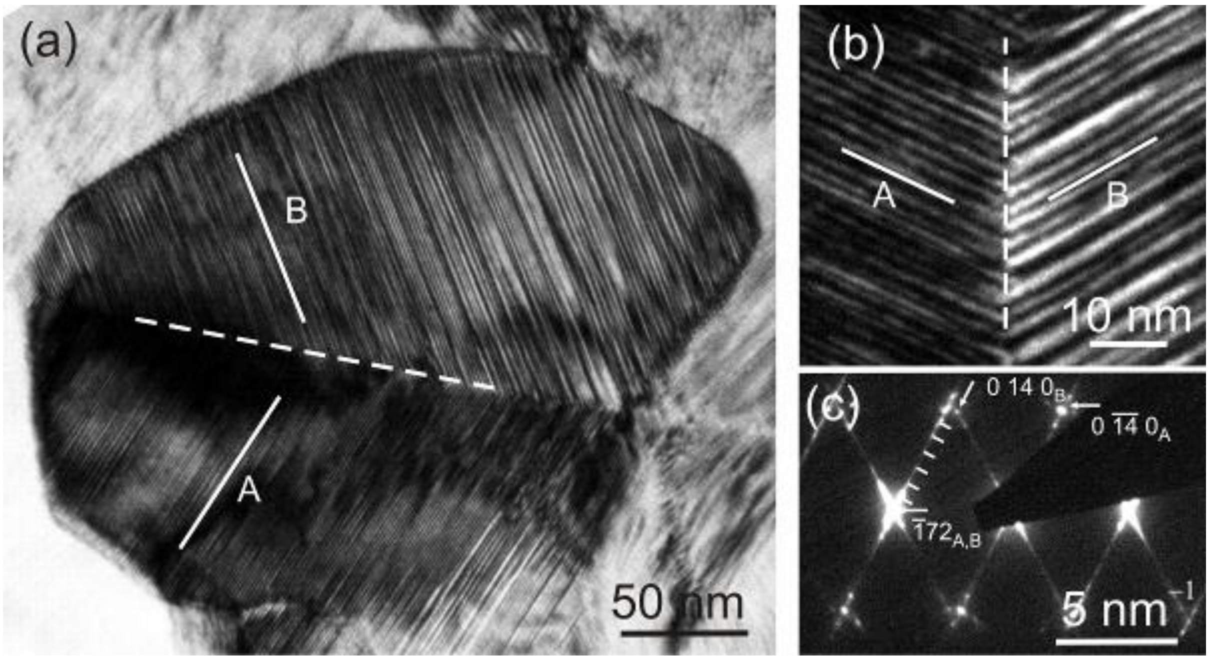

Figure 2. Ultrafine grained $Ni_{54}Mn_{25}Ga_{21}$ (a) TEM lattice fringe image of a grain containing two twin related variants A and B of 14M martensite. Solid and dashed lines indicate the (0 14 0) lattice planes and the junction plane of the variants, respectively, that all are in an edge-on orientation. (b) Detail of the junction plane that is almost straight and rather sharp. (c) Corresponding SAD pattern. Some of the satellite and main reflections are marked by lines and arrows, respectively. In agreement with a type I twin, variants A and B have a common $(\bar{1}72)$ junction plane and share a common zone axis. (**BD** = $[201]_A$ = $[\bar{2}0\bar{1}]_B$).