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## High strength and high ductility in a nanoscale superlattice of Ni<sub>2</sub>(Cr,Mo) deformable by twinning

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It is demonstrated that long-range ordering can be utilized to synthesize a nanoscale superlattice of  $Ni_2(Cr,Mo)$  in a Ni-18.6 at.% Mo-15.1 at.% Cr. The superlattice is distinguished by high strength and high ductility. This behavior has been correlated with the crystallography of the disorder-order transformation, which favors deformation by twinning on the {111} planes of the parent face-centered cubic structure.

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Recently, there has been an increasing interest in developing nanoscale materials combining high strength and high ductility. Although several synthesis techniques have been advanced to produce such materials [1], modifications of various plastic deformation processes appear to be the most viable (e.g. [2–7]). These processes are based upon producing an ultra fine grain structure on the nanoscale by high plastic strain rate favoring deformation by twinning. In this paper, we demonstrate an alternative approach where long-range ordering in certain alloys can be utilized to synthesize nanoscale superlattices that combine high strength and high ductility. We have chosen a Ni-Cr-Mo alloy as a model system with an approximate composition of Ni<sub>2</sub>(Cr,Mo). Alloys based upon this system have many important high- and lowtemperature applications in the petrochemical, chemical process, and power generation industries. Similar to the case of the Ni-Mo system, these alloys are known to undergo a series of long-range ordering reactions resulting in the formation of the closely-related DO<sub>22</sub> (Ni<sub>3</sub>Mo), D1<sub>a</sub> (Ni<sub>4</sub>Mo), and Pt<sub>2</sub>Mo-type superlattices [8]. Although these superlattices can co-exist during the early stages of ordering, it has been shown that the most stable ordered phase which forms in a given alloy is a sensitive function of chemical composition [8–11].

The alloy studied here had a composition of Ni–26.97 wt.% Mo–11.95 wt.% Cr corresponding to Ni–18.5 at.% Mo–15.1 at.% Cr. Bulk samples were processed into sheets about 1.5 mm in thickness, and then annealed at 1065 °C for 15 min followed by water quenching. Long-range ordering was induced by thermal aging at 700 °C. Transmission electron microscopy (TEM) and diffraction as well as high-resolution transmission electron microscopy (HRTEM) were used to characterize the microstructure. Thin-foil specimens were prepared by the jet polishing technique. Mechanical strength was determined from room-temperature tensile tests carried out on standard samples with 50.8 mm gage length. All crystallographic notations are given in terms of the parent face-centered cubic (fcc) structure of the ordered phases.

As shown in Figure 1a–c, the  $DO_{22}$ , D1a, and  $Pt_2Mo$ -type superlattices can directly be derived from the parent fcc lattice by minor atomic rearrangement on the  $\{420\}$  planes as shown in Figure 1a–c. Figure 1d shows a schematic of the (001) reciprocal lattice intersection illustrating the relative positions of the corresponding superlattice reflections. As can be seen, the characteristic reflections of the  $DO_{22}$  superlattice are located at  $\langle 1\ 1/2\ 0\rangle$ ,  $\langle 100\rangle$ , and  $\langle 1\ 1\ 0\rangle$  positions, and those of the  $D1_a$  and  $Pt_2Mo$ -type superlattices are located at  $1/5\ \langle 420\rangle$  and  $1/3\ \langle 420$  (or equivalently  $1/3\ \langle 220\rangle$ ), respectively. Although  $Ni_2Mo$  is not thermodynamically stable in the Ni–Mo system, it can stabilized by additions of Cr since  $Ni_2Cr$  is stable in the Ni–Cr system.

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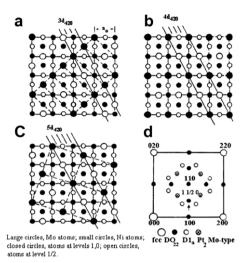


Figure 1. Schematics illustrating the atomic arrangements of various superlattices along  $\langle 001 \rangle$  direction and relative positions of respective superlattice reflections: (a) DO<sub>22</sub> superlattice, (b) D1<sub>a</sub> superlattice, (c) Pt<sub>2</sub>Mo-type superlattice, (d)  $\langle 001 \rangle$  reciprocal lattice intersection.

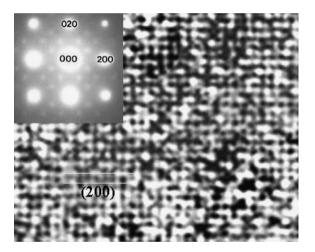
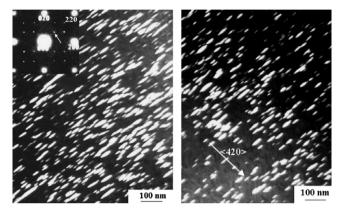


Figure 2. State of short-range order in the as-quenched condition illustrated by  $\langle 001 \rangle$  diffraction pattern and corresponding HRTEM image.

In the as-quenched condition, diffuse intensity maxima due to short-range order were observed at  $\langle 1\ 1/2\ 0\rangle$  positions similar to the case of Ni–Mo alloys [8–13]. Also, the corresponding HRTEM images resembled those obtained in the case of Ni4Mo as shown in the example of Figure 2. Using multi-slice image simulations, an image such as that shown in Figure 2 has been interpreted in terms of subunit cell clusters of the DO<sub>22</sub>,

D1<sub>a</sub>, and Pt<sub>2</sub>Mo-type superlattices using multi-slice image simulations [14]. A further evidence for the co-existence of these superlattices in the present case is provided by electron diffraction as shown below.

Figure 3 illustrates the  $\langle 001 \rangle$  electron diffraction pattern as a function of thermal aging time at 700 °C. As shown earlier, diffuse intensity maxima are observed at  $\{11/20\}$  positions in the annealed state (Figure 3a). During the early stages of thermal exposure (5 min at 700 °C), those intensity maxima became elongated along  $\langle 100 \rangle$  directions suggesting the onset of formation of DO<sub>22</sub> superlattice (Fig. 3b). With continued exposure, characteristic reflections of the three superalattices be-



**Figure 4.** Dark-field TEM images formed with  $1/3\langle 220\rangle$  superlattice reflections to illustrate the morphology of ordered particles as a function of aging time at 700 °C: (a) 24 h; the inset is a  $\langle 001\rangle$  diffraction pattern, (b) 100 h.

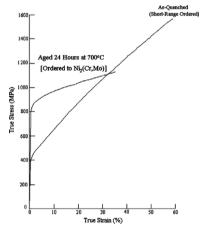


Figure 5. Comparative true stress-strain diagrams in the disordered and ordered states.

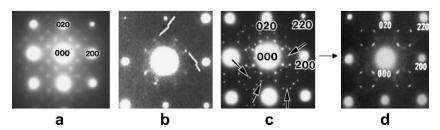


Figure 3. (001) selected-area diffraction patterns illustrating the transition from short-range order into long-range order during thermal aging at 700 °C; (a) as-quenched, (b) 5 min at 700 °C, (c) 15 min at 700 °C, (d) 8 h at 700 °C.

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