



Interfacial mixing of nickel vanadium multilayers induced by cold rolling

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Abstract—During the cold rolling of multilayers of Ni and V with an average composition $\text{Ni}_{70}\text{V}_{30}$, $\text{Ni}_{30}\text{V}_{70}$ and $\text{Ni}_{57}\text{V}_{43}$, deformation induces phase transformation and an interfacial mixing. After repeated cold rolling at room temperature, X-ray diffraction (XRD) and scanning transmission electron microscope (STEM) results demonstrate that between pure Ni and V layers a metastable fcc solid solution phase forms in $\text{Ni}_{70}\text{V}_{30}$, a metastable bcc solid solution phase forms in $\text{Ni}_{30}\text{V}_{70}$ and metastable fcc and bcc solid solution phases form in $\text{Ni}_{57}\text{V}_{43}$ with suppression of nucleation of intermetallic phases. At an intermediate stage of the reaction when both pure Ni and V still remain, a smoothly varying composition profile was determined from the electron energy loss spectrum (EELS) across the multilayers. The composition dependent effective interdiffusion coefficient that was calculated based on the obtained profile was found to increase monotonically from $1.2 \times 10^{-15} \text{ cm}^2/\text{s}$ to $1.6 \times 10^{-13} \text{ cm}^2/\text{s}$ with increase of Ni mole fraction. Compared to the stored energy due to dislocation and interfaces, the excess chemical free energy from the interfacial mixing is the largest portion of total stored energy from deformation, which represents a form of mechanochemical transduction.

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1. Introduction

Mechanical alloying (MA) is a well-known method to improve the properties and performance of materials [1]. The unique capability of MA is exemplified by several novel structures such as the extension of solid solubility in systems that are immiscible in equilibrium [2–4], the synthesis of metallic glass [5] and different kinetic pathways in solid solution formation [6]. These novel structures are not initiated by the usual thermally activated diffusion. Mechanical alloying has been widely applied for industrial use due to its practical simplicity. However, the governing mechanism can be complex compared to thermal activation [7]. For example, when the powder particles are impacted by the balls used in milling, it is expected that the cold welding between the powders initiates the alloying process. With continued ball milling, the powder particles become elongated and layered with a refined nanoscale microstructure. Due to the chaotic nature of ball milling different powder particles experience different levels of deformation and alloying so that it is difficult to relate the microstructural evolution directly to the deformation level or the energy input. On the other hand, the repeated cold rolling of multilayers allows for the detailed examination of interface reactions at each stage of deformation so that a clear measure can be obtained between the extent of interfacial

atomic scale mixing and the input of deformation work. In a prior work, the extent and character of the interfacial mixing was examined during deformation of multilayers in the isomorphous Cu–Ni system [6,8]. As a next step in the systematic study of deformation induced interfacial reactions, the Ni–V system was selected since the end members have different crystal structures and there are several intermediate alloy phases. In addition there are thermodynamic and kinetic data available for the analysis of the structural evolution during intense deformation.

2. Experimental methods

The 25 μm thick Ni and V elemental foils with a purity of 99.8% were cut and weighed separately to achieve the average compositions $\text{Ni}_{70}\text{V}_{30}$, $\text{Ni}_{30}\text{V}_{70}$ and $\text{Ni}_{57}\text{V}_{43}$ in atomic percent. Then, the Ni and V foils were stacked alternatively to form the sandwich array as a Ni/V/Ni/V structure with different initial areal size to achieve the desired composition. The cold rolling experiments were performed with a hand rolling mill. The time to accomplish a roll is estimated as 1 s and the multilayer foil was folded in half after each roll that reduced the sample thickness by 50%. The deformation level was described by the number of passes in which one pass consists of a roll and a fold. With the folding operation after each rolling pass no new reaction interfaces are created and all existing reaction interfaces experience the same number of passes. The phase

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transformation and structural evolution were monitored with a Bruker D8 Advance X-ray Diffractometer (XRD). The microstructure was examined with Zeiss LEO 1530-2 scanning electron microscope (SEM) and FEI Titan scanning transmission electron microscope (STEM) with a CEOS probe aberration corrector. The composition was analyzed from the electron energy loss spectrum (EELS) using the Gatan Digital Micrograph software. The STEM samples were prepared by Zeiss 1540XB cross beam focused ion beam (FIB) system with 30 kv milling followed by a 5 kv cleaning as the final step to minimize the surface contamination.

3. Results and discussions

3.1. Evolution of solid solution formation and composition profile

The XRD pattern for the as-rolled $\text{Ni}_{70}\text{V}_{30}$ multilayer sample after different passes can be monitored as in Fig. 1(a). During the initial 15–30 passes, only individual peaks corresponding to pure fcc Ni and bcc V appear in the XRD pattern. Above 35 passes, in the 40–50° 2θ range the Ni and V peaks start to convolute into each other which indicates the formation of a solid solution between the single phases. At 50 passes, an intermediate peak appears which indicates the solid solution phase becomes a major component in the sample. At 80 passes, the Ni and V peaks completely merge into one single peak which means the Ni and V single phases completely transform into the solid solution phase. The lattice parameters corresponding to Ni, V and solid solution phase with an average composition at different rolling passes are shown in Fig. 1(b). It is seen that both Ni and V have been consumed within 5 addition passes from 75 to 80 passes. No significant change of XRD patterns was observed from 80 to 90 passes. At 90 passes, the crystal structure of the solid solution was determined as fcc from the positions of diffraction peaks. From the evolution of the diffraction patterns, no significant change of Ni and V peak positions was observed when the pure Ni and V layers remain in the sample. It is also noted that since the fcc Ni has a lattice constant 0.352 nm [9] and bcc V has a lattice constant 0.304 nm [10], the solid solution phase has a larger lattice constant than pure Ni and the corresponding diffraction peak will shift to the smaller angle side compared to Ni peak according to Bragg's law [11]. This trend is shown for the Ni (200) peak around 50° and high angle peaks between 70° and 80°. If vanadium is assumed to be fcc, the corresponding lattice parameter as calculated from the hard sphere atom size of bcc is 0.372 nm. It is seen that the lattice parameter of the fcc solid solution is between that of fcc Ni and fcc vanadium. If Vegard's law [12] is applied on fcc Ni and fcc vanadium, the lattice parameter of the fcc solid solution is estimated as 0.358 nm for the $\text{Ni}_{70}\text{V}_{30}$ composition, which is very close to the XRD measured value that is 0.359 nm.

The XRD pattern for the as-rolled $\text{Ni}_{30}\text{V}_{70}$ multilayer sample after different passes can be monitored as in Fig. 2(a). It is seen that initially only fcc Ni and bcc V peaks appear in the XRD pattern. With further deformation, a solid solution forms and becomes a major component in a similar way as for the $\text{Ni}_{70}\text{V}_{30}$ sample. After 90 passes the Ni and V peaks completely merge into one single peak which indicates that the Ni and V pure components

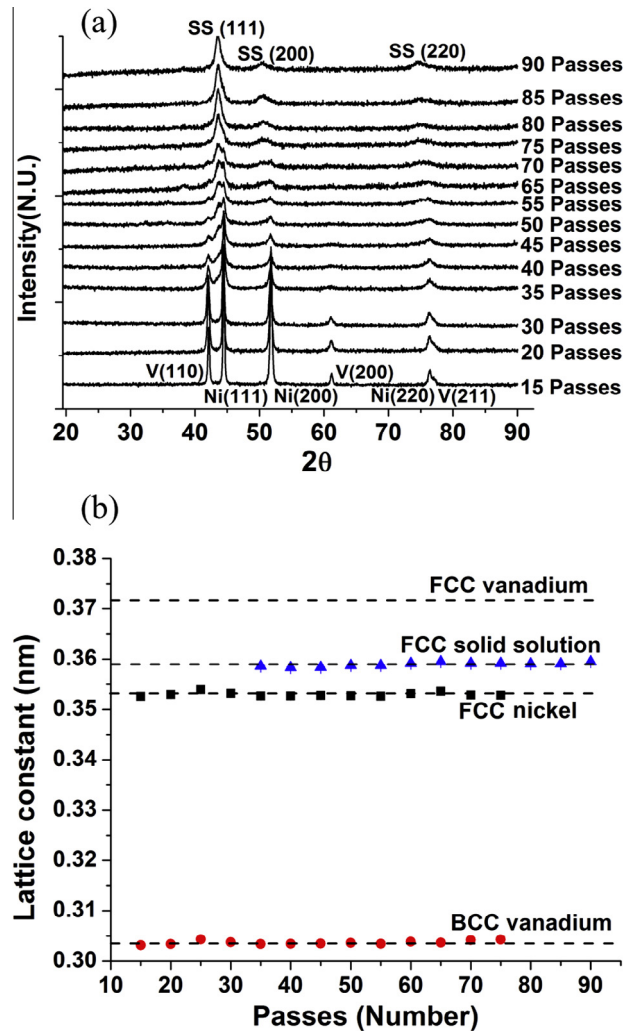


Fig. 1. (a) X-ray diffraction patterns for $\text{Ni}_{70}\text{V}_{30}$ multilayer. The pattern intensity was normalized for each pass. The corresponding diffraction peaks are labeled in the figures. The label SS stands for solid solution. (b) The lattice constants of Ni, V and solid solution phase with an average composition $\text{Ni}_{70}\text{V}_{30}$ at different rolling passes. The lattice constant of fcc vanadium is also labeled as a dashed line.

completely transform into the solid solution phase. In contrast to $\text{Ni}_{70}\text{V}_{30}$ sample, the crystal structure of the solid solution in $\text{Ni}_{30}\text{V}_{70}$ sample is determined as bcc from the positions of diffraction peaks. If nickel is assumed to be bcc, the corresponding lattice parameter is calculated from that of fcc Ni as 0.287 nm. This indicates that the lattice parameter of bcc solid solution phase is smaller than that of vanadium and the corresponding diffraction peak position will shift to the larger angle side compared to V peak. This trend is seen in Fig. 2(a) for V (200) peak between 60° and 65° and V (211) peak between 75° and 80°. The lattice parameters corresponding to Ni, V and solid solution phase with an average composition at different rolling passes are shown in Fig. 2(b). It is seen that the lattice parameter of bcc solid solution is between that of bcc Ni and bcc vanadium. If Vegard's law [12] is applied on bcc Ni and bcc vanadium, the lattice parameter of the bcc solid solution is estimated as 0.299 nm for the $\text{Ni}_{30}\text{V}_{70}$ composition. It is seen that the XRD measured value which is 0.296 nm is in good agreement with the estimated value from Vegard's law.

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