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# In situ production of bulk intermetallic-based nanocomposites and nanostructured intermetallics by high-pressure torsion

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Micropowder mixtures of Al–50 mol.% Ni were severely deformed by high-pressure torsion and $Al_3Ni_2$/Ni nanocomposites were produced. The hardness increased with straining, as a consequence of nanograin formation and high-strain-induced solid-state reactions, and saturated to a steady-state level, 920 Hv. The reactions were completed and nanostructured AlNi intermetallics were produced by subsequent annealing. The formation of nanostructured intermetallics was feasible in this process at significantly low temperatures because of the rapid diffusion and short diffusion paths resulting from intense shearing.


*Keywords:* Ultrafine-grained microstructure; Severe plastic deformation (SPD); High-pressure torsion (HPT); Mechanical alloying

Intermetallic materials, which have characteristics of both metals and ceramics, exhibit unique physical, chemical and mechanical properties especially in nanostructured form. Numerous routes for the production of intermetallics from their elemental constituents have been developed, including casting, thermomechanical processing, reactive sintering, mechanical alloying, physical vapor deposition and electrodeposition [1]. Despite this plethora of methods for production of intermetallics [1] and intermetallic-based composites [2], fabrication of bulk nanostructured intermetallics and their nanocomposites is still a challenging task.

Nanostructured intermetallics and their nanocomposites are generally produced by two-step processes: the first step involves synthesis of nanopowder intermetallics from their elemental constituents by methods such as chemical synthesis [3], high-energy ball milling [4], inert gas condensation [5] and laser vaporization condensation [6]; and the second step is the consolidation of nanopowders into bulk forms [1,2]. However, high temperatures need to be used in the second step to achieve a full consolidation and retaining nanograins is difficult when processing at high temperatures.

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It has been documented that application of high-temperature compression to elemental nanopowders can be used for the in situ production of bulk nanostructured intermetallics [7], but the starting materials need to be in the form of nanopowders, which are in general not easily fabricated. Application of severe plastic deformation (SPD) methods [8,9] to coarse-grained intermetallics is another approach for production of bulk nanostructured intermetallics [10,11]. However, the starting bulk intermetallics need to be produced by casting at extremely high temperatures and this is a challenging task. The SPD process is also applicable for cold consolidation of metal-based nanocomposites with large fractions of intermetallics or ceramics [12,13], but the starting intermetallics or ceramics should be in the form of nanopowders.

In this study, and for the first time, bulk intermetallic-based nanocomposites and nanostructured intermetallics in the Al–Ni system are generated from elemental micropowder mixtures by high-pressure torsion (HPT). Among different SPD methods, HPT, in which a disc is subjected to high pressure and simultaneous torsional straining, was selected for this study because it has several advantages: (i) HPT has ability to impose extremely large shear strains, $\gamma$ ($\gamma = 2\pi rN/h$, where $r$ is the distance from disc center, $N$ is the number of revolutions and $h$ is the disc thickness) [14]; (ii) HPT provides a unique opportunity for consolidation of different kinds of

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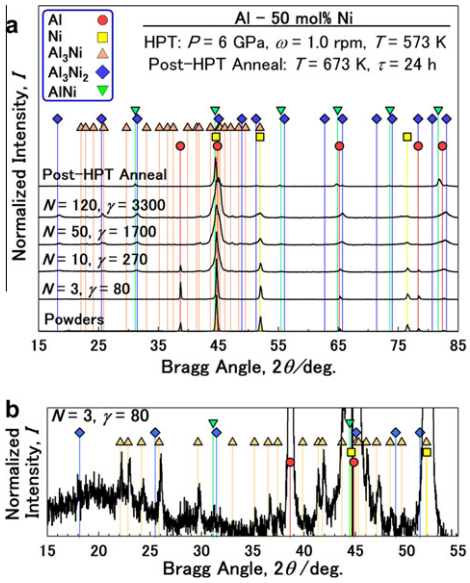


**Figure 1.** XRD profiles for (a) samples processed by HPT at 573 K for $n = 3$, 10, 50 and 120 turns and sample post-HPT annealed at 673 K for 24 h including as-received Al/Ni micro-powder mixtures, and (b) sample processed for $n = 3$ in enlarged scale to check $Al_3Ni$ formation.

materials even including hard and less ductile intermetallics and ceramics [15]; and (iii) HPT can be used for controlling phase changes in metals [16,17] and ceramics [18].

Al (99.99%) powders with <75 μm particle sizes were mixed with 50 mol.% Ni (99.99%) powders with <150 μm particle sizes. As reference materials, discs of pure Al (99.99%) and pure Ni (99.99%) 10 mm in diameter and 0.8 mm thick were also used. HPT was conducted at 573 K to consolidate the powder mixtures to discs 10 mm in diameter and 0.8 mm thick under a pressure of 6 GPa. Shear strain was introduced through rotations for either $n = 3$, 10, 50 or 120 revolutions with a rotation speed of $\omega = 1.0$ rpm. The sample processed for $n = 50$ was subsequently annealed at 673 K for 24 h.

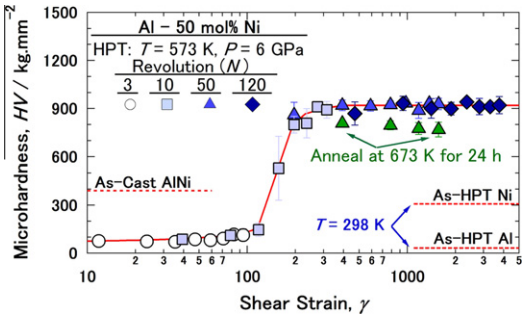


**Figure 3.** Vickers microhardness plotted against shear strain for samples processed by HPT at 573 K for $n = 3$, 10, 50 and 120 and sample post-HPT annealed at 673 K for 24 h including hardness levels for as-HPT pure Al and Ni and as-cast AlNi.

The HPT-processed discs were first polished to a mirror-like surface and Vickers microhardness was measured with an applied load of 200 g for 15 s along the radii at eight different radial directions. X-ray diffraction (XRD) analysis was performed using Cu $K_\alpha$ radiation. Scanning electron microscopy (SEM) at 20 kV was used for microstructural observations. The sample density was determined by Archimedes' principle. Finally, 3 mm discs were cut at 3.5 mm from the disc center and thinned with an electrochemical polisher (10% $HSO_4$, 10% $HNO_3$, 80% $CH_3OH$) and examined by transmission electron microscopy (TEM) at 300 kV.

XRD analysis, as shown in Figure 1, confirms that the $Al_3Ni$ intermetallic is formed at the early stages of straining ($\gamma = 80$) but transforms to an $Al_3Ni_2$ intermetallic on further straining and to an AlNi intermetallic by subsequent annealing at 673 K for 24 h. The material at large strains ($\gamma > 270$) consists of ~50 mol.% (84 vol.%) $Al_3Ni_2$ and ~50 mol.% Ni after HPT and of ~100% AlNi after annealing.

The SEM results are shown in Figure 2a. It is apparent that Al and Ni phases are distinctive after compression, but the two phases are elongated significantly in the shear direction and their thickness is reduced with

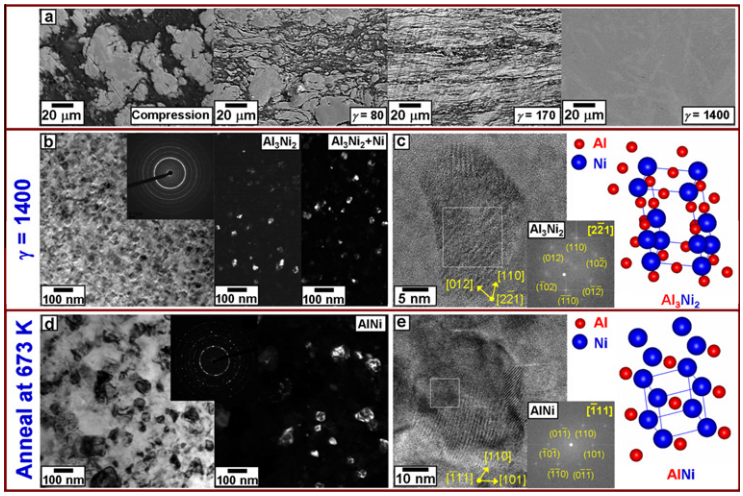


**Figure 2.** Microstructures of samples. (a) SEM micrographs after compression and after HPT for strains of $\gamma = 80$, 170 and 1400. (b) TEM bright-field image, SAED pattern and dark-field images corresponding to $Al_3Ni_2$ and $Ni + Al_3Ni_2$ phases for sample processed for $\gamma = 1400$. (c) High-resolution image and corresponding FFT analysis from square region for $Al_3Ni_2$. (d) TEM bright-field image, SAED pattern and dark-field images corresponding to AlNi phase for sample subjected to post-HPT annealing at 673 K for 24 h. (e) High-resolution image and corresponding FFT analysis from square region for AlNi.